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Technical note: Improving modeling of coagulation, curd firming, and syneresis of sheep milk

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ABSTRACT

The importance of milk coagulation properties for milk processing, cheese yield, and quality is widely recognized. The use of traditional coagulation traits presents several limitations for testing bovine milk and even more for sheep milk, due to its rapid coagulation and curd firming, and early syneresis of coagulum. The aim of this technical note is to test and improve model fitting for assessing coagulation, curd firming, and syneresis of sheep milk. Using milk samples from 87 Sarda ewes, we performed in duplicate lactodynamographic testing. On each of the 174 analyzed milk aliquots, using 180 observations from each aliquot (one every 15 s for 45 min after rennet addition), we compared 4 different curd firming models as a function of time (CF_t, mm) using a nonlinear procedure. The most accurate and informative results were observed using a modified 4-parameter model, structured as follows: $CF_t = CF_P \times \left(1 - e^{-k_{CF}\left(t - RCT_{eq}\right)}\right) \times e^{k_{SR} \times \left(t - RCT_{eq}\right)}$, where t is time, RCT_{eq} (min) is the gelation time, CF_P (mm) is the potential asymptotical CF at an infinite time, k_{CF} (%/min) is the curd firming rate constant, and k_{SR} (%/min) is the curd syneresis rate constant. To avoid nonconvergence and computational problems due to interrelations among the equation parameters, CF_P was preliminarily defined as a function of maximum observed curd firmness (CF_{max}, mm) recorded during the analysis. For this model, all the modeling equations of individual sheep milk aliquots were converging, with a negligible standard error of the estimates (coefficient of determination >0.99 for all individual sample equations). Repeatability of the modeled parameters was acceptable, also in the presence of curd syneresis during the lactodynamographic analysis.

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Technical Note

Milk coagulation can be measured by a wide range of mechanical, vibrational, ultrasonic, thermal, and optical methods (O'Callaghan et al., 2002; Klandar et al., 2007). Among those, the monitoring with time of curd firmness (**CF**, mm) of renneted milk samples maintained at a fixed temperature is frequently used to assess the coagulation process (Barbano and Lynch, 2006). Although it presents several limitations, the lactodynamography analysis is extensively used to assess coagulation and curd firming process (Annibaldi et al., 1977; McMahon and Brown, 1982), as it is simple and fast, and it could be used to test contemporarily several milk samples (usually 10). In bovine milk, the major limitations of lactodynamography are related to the traditional single point traits measured by the computerized instruments and include (1) the moderate repeatability of all the traits, (2) the high incidence of noncoagulating samples (rennet coagulation time, **RCT**, not measurable within 30 min from rennet addition) and coagulated samples not attaining a curd firmness of 20 mm ($\mathbf{k_{20}}$, min), and (3) the low-informative value of the curd firmness traditionally measured at the end of analysis ($\mathbf{a_{30}}$, mm). To solve those limits, Bittante (2011) presents a 3-parameter model that used all the CF measures of a sample (one every 15 s) as a function of time ($\mathbf{CF_t}$, mm) over the traditional 30-min analysis interval. The 3 estimated parameters are the RCT from the model equation ($\mathbf{RCT_{eq}}$, min), the potential asymptotic curd firmness ($\mathbf{CF_P}$, mm), and the instant rate constant of curd firming ($\mathbf{k_{CF}}$, %/min). That model reduces the limitations of lactodynamography (except for the incidence of noncoagulating samples), as the parameters are estimated using all the information (120 observations/sample) of the analysis. Moreover, the high presence of bovine milk samples not coagulating within 30 min suggests to extend the interval analysis to 60 or even to 90 min after rennet addition (Cipolat-Gotet et al., 2012). The extension of the analysis of bovine milk indicates CF reduction after the attainment of a maximum value, interpreted by Bittante et al. (2013) as a sign of the beginning of syneresis. Hence, those authors propose a new equation model introducing a fourth parameter (**k_{SR}**, %/min), which describes the instant rate constant of syneresis. A limitation of those CF_t models is the presence of milk samples whose equation does not converge. Nonconverging samples (i.e., very late coagulating samples), provide a few number of available observations to permit a precise estimation of modeled parameters. In those cases, the equation cannot estimate modeled parameters or outliers. Sheep milk presents a pattern of coagulation and curd firming very different from bovine milk (Bencini, 2002; Bittante et al., 2014; Ferragina et al., 2017) because the RCT is usually shorter, the curd firming more rapid, the maximum CF greater, and the syneresis appears earlier (often before 30 min after rennet addition). As a consequence, traditional coagulation traits developed on bovine milk are often not so informative for sheep milk. The aim of this technical note was to improve the CF_t model fitting for assessing coagulation, curd firming, and syneresis and to test these improvements to model individual sheep milk samples.

To perform the present study, we used data from the database previously described in Pazzola et al. (2014). Among the animals sampled for that study (1,121 ewes from 23 flocks of Sarda sheep located in the island of Sardinia, Italy), 2 to 13 ewes from 18 farms were selected for a total of 87 ewes. Selected ewes were from the second to the seventh month of lactation (DIM 150 ± 47 d) and had a daily milk yield of 1.53 ± 0.78 kg/d. After the collection, milk samples were divided into 2 aliquots (50 mL each) and kept at 4°C (without preservative). Traditional single-point traits (RCT, k_{20}, a_{30}, a_{45}, and a_{60}) and all the CF measures (240 for each milk aliquot, 1 every 15 s) were measured using a lactodynamograph (Formagraph, Foss Italia, Padova, Italy) and prolonging the test length until 60 min. Two aliquots of each individual milk sample (10 mL each aliquot) were heated at 35°C and mixed with 200 μL of the calf rennet solution [Hansen Naturen Plus 215 (Pacovis Amrein AG, Bern, Switzerland), with 80 ± 5% chymosin and 20 ± 5% pepsin and 215 international milk clotting units/mL; diluted to 1.2% (wt/ vol) in distilled water for the achievement of 0.0513 international milk clotting units/mL of milk]. For all the milk aliquots (n = 174), RCT and k_{20} were achievable within the first 45 min of the lactodynamographic analysis. Therefore, to compare different CF_t models, only the first 45 min (180 observations per aliquot) were used to estimate model parameters, as this is considered an adequate time interval to attain syneresis in individual ewe milk samples (Vacca et al., 2015). We tested 4 different nonlinear models for curd firming (CF_t, mm). The first model (**$3Par_{fixed}$**) was the 3-parameter asymptotic model proposed by Bittante (2011):

$$CF_t = CF_P \times \left(1 - e^{-k_{CF}\left(t - RCT_{eq}\right)}\right),$$

where CF_t is CF at time t (mm); CF_P is the asymptotical potential value of CF at an infinite time (mm); k_{CF} is the curd firming rate constant (%/min) and describes the shape of the curve from coagulation time to infinity (velocity of curd firming); and RCT_{eq} has the same meaning as the traditional RCT trait. A second model (**$3Par_{variable}$**) had the same equation as above but was based on a different and variable number of observations (≤180). In this second model, the observed maximum CF value (**CF_{max}**, mm) for each analyzed aliquot was used to measure the corresponding time after rennet addition (**t_{max}**, min, ≤45 min). Only the CF observations between the rennet addition and t_{max} were used for the estimation of the parameters of $3Par_{variable}$ model. In the case of aliquots presenting tendency to asymptote, the first value of CF_{max} was used if more than one maximum value was recorded during the single analysis. In this way, the potential decreasing phase of the CF_t curve was excluded from calculations, the number of observations was retained, and the corresponding degrees of freedom were different among the analyzed aliquots. The third was a 4-parameter model (**$4Par_{free}$**) previously proposed for bovine milk by Bittante et al. (2013), characterized by prolonging the duration of the lactodynamographic test and including the aforementioned parameters together with k_{SR} (%/min), the instant rate constant of syneresis, as follows:

$$CF_t = CF_P \times \left(1 - e^{-k_{CF}\left(t - RCT_{eq}\right)}\right) \times e^{k_{SR} \times \left(t - RCT_{eq}\right)}.$$

The $4Par_{free}$ model was proposed as a large percentage of individual milk samples showed a decreasing phase of CF_t, sometimes within 30 min after rennet addition (Vacca et al., 2015). In the first part of the curve, the effect of k_{CF} prevails over k_{SR}, and CF_t tends to increase until t_{max}. At t_{max} the effects of k_{CF} and k_{SR} are equal and opposite in sign. At t_{max}, the effects of these 2 traits are equal and opposite. After t_{max}, the effect of syneresis becomes more forceful than k_{CF}, and CF_t curve tends to decrease toward a null value. The decrease of CF is apparent and is due to the increasing