

# Potential estimation of titratable acidity in cow milk using mid-infrared spectrometry

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# Context

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- Interest of MIR spectrometry
  - Review of Soyeurt H., ICAR 2010
  - The technological abilities of milk

# Context

- Estimation of technological abilities
  - Finer and more accurate estimations
    - ✓ Better support to farmers/producers
    - ✓ Finer management of the herd
    - ✓ Improve transformation yields

# Context

- Cheese making process
  - Global cheese yield
  - Milk coagulation properties
    - ✓ up to 40 % of variation among cows (Ikonen *et al.*, 2004)
- Factors influencing milk coagulation kinetics
  - Coagulation enzyme
  - Protein and calcium contents in milk
  - Temperature
  - Acidity (O'Callaghan *et al.*, 2001)

# Context

## ■ Titratable acidity (TA)

- TA influences all phases of milk coagulation
- Developed acidity results from bacterial activity
  - ✓ Lactic acid
  - ✓ Collection, transportation, and transformation of milk
- Fresh milk
  - ✓ Some components: carbon dioxide, citrates, casein, albumin/globulin and phosphates
  - ✓ Buffer action

# Objective

- To investigate the potential use of MIR spectrometry in order to predict TA
  - TA recorded as Dornic degree
  - Walloon Region of Belgium
  - Multibreed

# Material and methods

## ■ Sampling

- Walloon Region of Belgium
- Large variability: several criteria
  - ✓ Milk sampling: individual or bulk milk
  - ✓ Breed: Dual Purpose Belgian Blue, Holstein, Red-Holstein, Montbeliarde and Jersey
  - ✓ Time of sampling: morning milking, evening milking or mix of 50 % morning & 50 % evening milk samples

# Material and methods

## ■ Analysis

- 225 samples
- Milk Lab (Comité du Lait, Battice, Belgium)
  - ✓ MIR Foss MilkoScanFT6000 spectrometer
  - ✓ Analysed traits: fat, protein, free fatty acid (FFA), urea, lactose, dry matter (DM), somatic cell count (SCC) and pH
  - ✓ SCC → Somatic Cell Score (SCS)

# Material and methods

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## ■ Analysis

- Titratable acidity
  - ✓ Recorded as Dornic degree (D°)
  - ✓ 0.1 N NaOH solution
  - ✓ Consumption of NaOH to shift the pH value from 6.6 to 8.4 (phenolphthalein)

# Material and methods

- Calibration procedure
  - First derivative pretreatment
  - Partial least square regressions
  - 22 outliers
  - Statistical parameters
    - ✓ Mean and standard deviation (SD)
    - ✓ Standard error of calibration (SEC)
    - ✓ Calibration coefficient of determination ( $R^2_C$ )

# Material and methods

- Calibration procedure
  - Cross-validation
    - ✓ To determine the number of factors
    - ✓ To assess the accuracy of equation
    - ✓ Partitioning randomly the calibration set: 102 groups
  - Statistical parameters to assess the accuracy
    - ✓ Standard error of cross-validation (SECV)
    - ✓ Calibration coefficient of determination ( $R^2_{CV}$ )
    - ✓  $RPD = SD / SECV$

# Results

## ■ Characterization of the samples

| Trait                   | Mean  | SD    |
|-------------------------|-------|-------|
| Fat (%)                 | 3.88  | 1.03  |
| Protein (%)             | 3.49  | 0.52  |
| FFA (mmol/100 g of Fat) | 5.63  | 8.62  |
| Urea (g/100 mL)         | 0.023 | 0.011 |
| Lactose (g/100 mL)      | 4.85  | 0.35  |
| DM (%)                  | 12.66 | 1.25  |
| SCS                     | 3.31  | 1.90  |
| pH                      | 6.69  | 0.09  |
| TA (D°)                 | 16.27 | 2.27  |

\* FFA = Free Fatty Acid; DM = Dry matter; SCS = somatic cell score;  
D° = Dornic degrees.

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| Lactose (g/100 mL)      | 4.85                                   | 0.35        |
| DM (%)                  | 12.66                                  | 1.25        |
| SCS                     | 2.21                                   | 1.00        |
| pH                      | <b>Coefficient of Variation = 14 %</b> |             |
| <b>TA (D°)</b>          | <b>16.27</b>                           | <b>2.27</b> |

\* FFA = Free Fatty Acid; DM = Dry matter; SCS = somatic cell score;  
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# Results

## ■ Observed correlations among milk components

|         | Fat                | FFA     | Protein | Urea    | Lactose             | DM                 | SCS                | pH                  |
|---------|--------------------|---------|---------|---------|---------------------|--------------------|--------------------|---------------------|
| TA (D°) | 0.04 <sup>NS</sup> | 0.13*   | 0.39*** | 0.18**  | 0.21**              | 0.26***            | -0.16*             | -0.32***            |
| Fat     | -                  | 0.41*** | 0.42*** | 0.13*   | -0.19**             | 0.89***            | 0.18**             | -0.18**             |
| FFA     |                    | -       | 0.68*** | 0.41*** | -0.17**             | 0.50***            | 0.04 <sup>NS</sup> | -0.38***            |
| Protein |                    |         | -       | 0.30*** | -0.07 <sup>NS</sup> | 0.69***            | 0.10 <sup>NS</sup> | -0.26***            |
| Urea    |                    |         |         | -       | 0.18**              | 0.25***            | -0.18**            | -0.01 <sup>NS</sup> |
| Lactose |                    |         |         |         | -                   | 0.11 <sup>NS</sup> | -0.40***           | 0.66***             |
| DM      |                    |         |         |         |                     | -                  | 0.07 <sup>NS</sup> | -0.06 <sup>NS</sup> |
| SCS     |                    |         |         |         |                     |                    | -                  | -0.19**             |

FFA = Free Fatty Acid; DM = Dry matter; SCS = somatic cell score; D° = Dornic degrees.

\* = *P*-value < 0.05; \*\* = *P*-value < 0.01; \*\*\* = *P*-value < 0.001; NS = non significant.

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| Fat     | -                        | <b>0.41***</b> | <b>0.42***</b> | <b>0.13*</b>   | <b>-0.19**</b>            | <b>0.89***</b>           | <b>0.18**</b>            | <b>-0.18**</b>            |
| FFA     |                          | -              | <b>0.68***</b> | <b>0.41***</b> | <b>-0.17**</b>            | <b>0.50***</b>           | <b>0.04<sup>NS</sup></b> | <b>-0.38***</b>           |
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| Urea    |                          |                |                | -              | <b>0.18**</b>             | <b>0.25***</b>           | <b>-0.18**</b>           | <b>-0.01<sup>NS</sup></b> |
| Lactose |                          |                |                |                | -                         | <b>0.11<sup>NS</sup></b> | <b>-0.40***</b>          | <b>0.66***</b>            |
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# Results

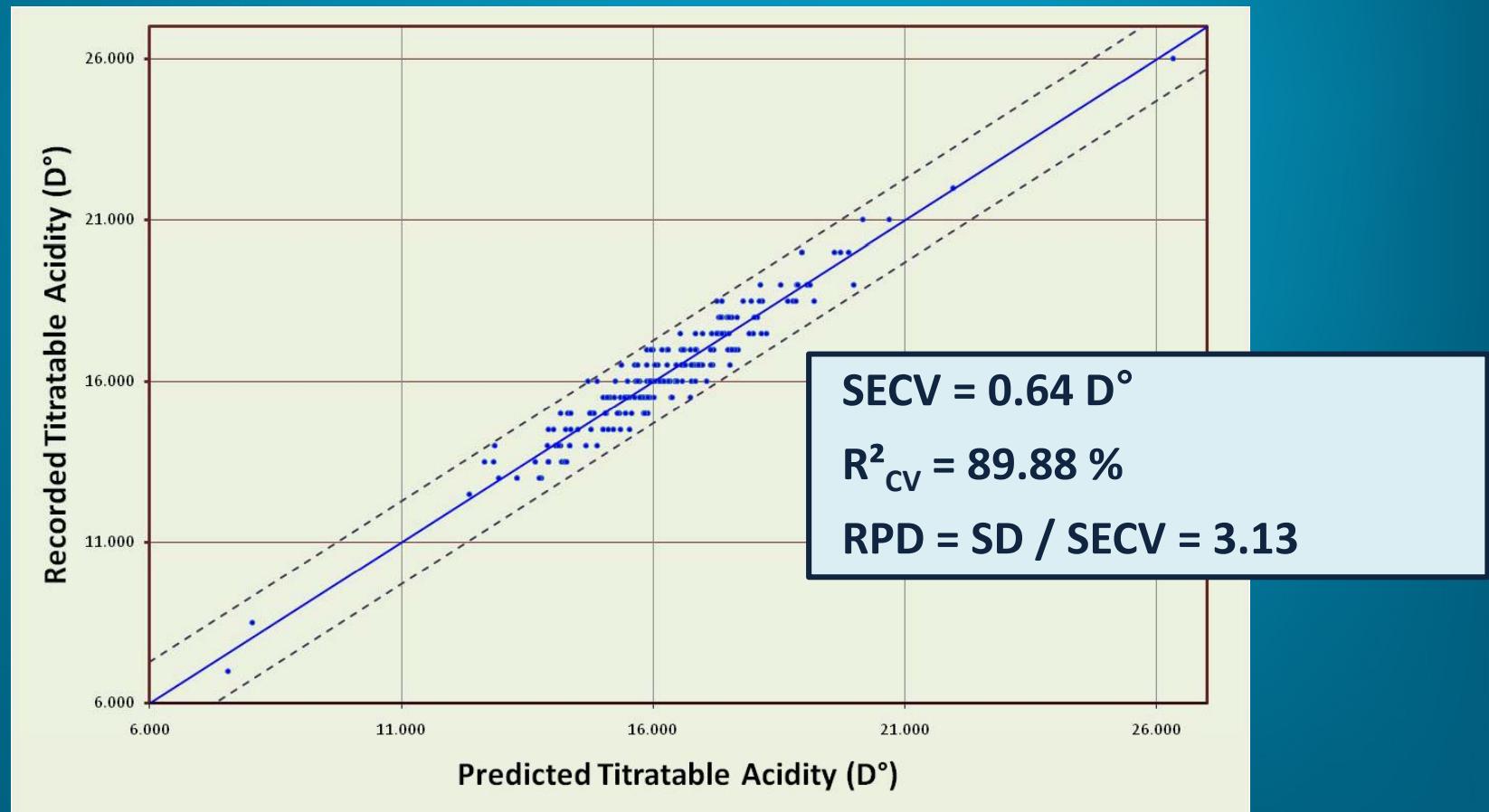
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## ■ Statistical parameters

- 10 factors
- Mean  $\pm$  SD =  $16.22\text{ D}^\circ \pm 2.01$  ( $n = 203$ )
- SEC =  $0.56\text{ D}^\circ$
- $R^2_C = 92.25\%$

# Results

## ■ Cross-validation



# Conclusions

- $R^2_C$  and  $R^2_{cv}$ 
  - High and around 90 %
  - Higher than observed correlations (max 39 %)
- $RPD > 2$

→ Feasibility of TA prediction in bovine milk from MIR spectrum

→ Calibration equation: good predictor and usable in most applications (including research)

# Perspectives

- Validation with new set of samples
- Use of this equation
  - Walloon Database: 900,000 spectra
  - Study of TA variability in the Walloon dairy cattle
    - ✓ Detection of potential effects of breed, season, DIM...
  - Development of a genetic evaluation
  - TA breeding values + others traits = a new economic index for cheese making abilities ?

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- National Fund for Scientific Research



# Thank you for your attention

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- Milk Committee of Battice
- Walloon Breeding Association (AWE asbl)
- Walloon dairy breeders

