A SIMPLE METHOD FOR OBSERVING RENNET COAGULATION IN GOAT MILK

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ABSTRACT

Renneting properties of goat milk were examined by a vibration viscometer in different renneting condition like CaCl₂ concentration, enzyme concentration and temperature. We observed markedly differences in the cutting time and the curd viscosity. All the increase of temperature, enzyme and CaCl₂ concentration caused a decrease in the cutting time and also in the curd viscosity. Our results related CaCl₂ addition was contrary with the literature can explain that we used raw goat milk. The use of heat treated goat milk could give different results partly which needs further experiments. The effect of the increase of renneting temperature on the cutting time equalized at 38°C but the curd viscosity was markedly lower. Therefore the increase of renneting temperature more than 38°C does not pay making cheese from raw goat milk. We explained a great variety in viscosity (from 80mPas tor 600mPas) and cutting time with different renneting condition, therefore many experiments is needed to clear the acceptable parameters of renneting. We would like to continue the work related to the cutting time predicting model.

1. INTRODUCTION

Curd is usually cut in cheese vats after a predetermined, enzymatic reaction time has elapsed or when the operator judges the curd suitable for cutting based on a subjective evaluation of textural and visual properties of the curd. Cutting the curd after a predetermined time is questionable because there are many-many factors (such properties of raw milk, heat treating, enzyme concentration, Ca⁺⁺ concentration, clotting temperature, etc. which affect the coagulation (coagulation time, and curd firmness) of milk. Mainly the curd firmness could cause a variation in the optimum cutting time. But the optimum time of cutting is related to the working of curd, curd loss in whey, cheese yield, composition and quality of cheese, and at the end, fundamentally, to the economical cheese making.

Cutting the curd based on the subjective judgement of the operator can be accurate and acceptable if the observation and evaluation of milk gel is done properly (Hori, 1985). But, if the gel is too firm at cutting time, syneresis will be retarded, resulting high moisture and acidity in cheese. If the curd is cut too soft then cheese yield will be decreased as a result of increased loss of fat and curd fines in the whey (Hori, 1985; Payne, Hicks, & Shen, 1993a). These reasons suggest using objective methods to determinate the optimal cutting time in cheese making.

Numerous methods have been developed for observing of milk clotting. First methods based on destructive process like mechanical curd firmness testers have been proposed to measure milk coagulation parameters in cheese vats (Richardson, Okigbo, Thorpe, 1985; Ustunol, Hicks, 1990; McMahon, Brown, 1982). But these instruments are not practical moreover are not optimal in an automated on-line instrument. So, later have been several devices and instruments have been employed for monitoring coagulation parameters and/or used to determine optimum firmness for cutting.

Several methods based on optical properties of milk have also been used to follow coagulation (Guthy, Novak 1977; Hardy, Fanni 1981; McMahon, Brown, Ernstrom 1984a). More recently, changes in diffuse reflectance during cow's milk coagulation were

monitored using a fibre optic probe (Payne et al.1990). Scher, Hardy (1993) studied the evolution of casein micelle mean size and turbidity after adding rennet. They found that both turbidity and mean size of casein initially decreased after adding rennet and was followed by an increase in both turbidity and particle size. Eleya et al. (1995) studied the acid coagulation of milk from cows, goats and sheep at various temperatures also by a turbidimetric method based on light reflection. Dybowska, Fujio (1996a,b) used a colorimeter for monitoring the acid-induced milk gelation. McMahon, Brown (1990) measured changes in light scattering at 600 nm in coagulating milk.

More authors using different methods found that the changes of observing properties was describable with several and typical curve and they have found that the inflexion point of these curves was correlate with flocculation time of milk and cutting time of curd (Korolczuk, Maubois, Loheac 1986, Payne et al. 1993b, Eleya et al., 1995), Lochte et al. 1998, Castillo et al. 2000, Castillo et al. 2002. So the inflexion point of curves can be use to determinate of the accurate cutting time. Mentioned methods are adequate but the purchase of these instruments is very costly, so simpler and cheaper solutions also can be acceptable.

Cow's milk has been studied more extensively than goat's milk because of its larger commercial importance. A recent increase in consumption of dairy products (especially cheese) from goat milk has motivated further research into goat's milk processing.

Our aim was to develop a simple method for determining cutting time of curd from goat milk using a commercial vibration viscometer.

2. Material and Methods

Raw goat bulk milk from Hungarian Native white variety (earlier Hungarian White) goats was used for experiments. The rennet coagulation was investigated in goat milk with different enzyme (from 5.0 to 30.0 μ l/100g milk), Ca⁺⁺ concentration (0.34, 0.68, 1.02, 1.36, 1.70mmol), and renneting temperature (30, 34, 38, 40, 42, 44, 46°C). Crystalline CaCl₂ (REANAL, n:16381-1-01-38) and chimosin-pepsine enzyme mixture (Caglio Clerici Italy, 14500) were used in experiments. 10% solutions were made from CaCl₂ and from enzyme than the solutions were conditioned to 10 minutes at room temperature before using. The constant renneting temperature was ensured with a Memmert U-200 water bath (Germany). All experiments were repeat five-fold. The monitoring system is demonstrated in Fig. 1.



Fig. 1. Gelation properties measuring system

3. RESULTS

Similar curves were described by viscometer in every experiment. A typical curve is demonstrated in Fig. 1.



Fig. 1. Typical curves recorded by vibrating viscometer

Using the vibration viscometer we observed similar recorded curves as described in literature (Castillo et al. 2000, Castillo et al. 2002), so some specific point also could be identify. The *Flocculation point* is the first then the linkage among casein micelles begins. K^{20} or E^{30} as the *Coagulation time* which correlated the cheese yield due to the importance of curd firmness at cutting (Aleandri et al. 1989) are marked points, represent the distance in mm between the incriminate point of curve and the "X" axis in a millimetre paper measured by Formagraph. The inflexion point of curve correlate with the *Cutting time*, so knowing the time at inflexion point (Castillo et al. 2000, Castillo et al. 2002) the cutting time is predictable. Finally, the peak at maximum viscosity was detectable simply with SV-10 viscometer and it is seems to correlate also with the cutting time from our preliminary experiments. We think that the real cutting time can be predicted using this viscometer. Therefore we investigated the correlation between the peak time and the inflexion point of curves. Temporarily we use time at the peak as cutting time.

3.1. Enzyme concentration

Increase of enzyme concentration resulted shorter maximum peak time (cutting time). Differences were remarkable resulting dramatically difference in cutting time, confirming that the selection of the optimal enzyme concentration has great importance in the GMP (Good Manufacture Practice) of cheeses. The shortest cutting time, 18.5 min. was observed at the highest concentration (300µl enzyme solution) versus 93 min. cutting time at the smallest concentration (75µl enzyme solution) (Fig. 2.).



Fig. 2. The effect of enzyme concentration on the renneting

We found, that the curd viscosity at cutting time was also affected by different enzyme concentration. Higher enzyme concentration affected a decrease in the curd viscosity.

Results from the observation of the renneting of goat milk using different enzyme concentration confirm the results of Walstra (2003) partly but confirm fully the results of Nájera (2003) observing the renneting of cow milk. Our observing trend in the changing of the cutting time and in viscosity of curd agrees their results but is contrary related to curd firmness with results of Bencini (2002) investigating sheep and cow milk. Szalai (2008) also observed the increase of curd firmness up to a critical point (concentration) besides the cutting time decrease, but the further increase of enzyme concentration caused the change of the trend. Our opinion is the trend may be turn after a critical enzyme concentration.

We demonstrate the changing of the curd viscosity (curd firmness) and cutting time depending on enzyme concentration (Fig. 3.).



Fig. 3. Correlation of enzyme concentration with viscosity and cutting time

Both the cutting time and viscosity represents a decreasing trend in Fig. 3., but the enzyme concentration has markedly greater effect on the cutting time. This finding agrees with cited literature. As can be observe, the biggest changing occurred from 75μ l to 150μ l than the changing was smaller.

The published contrary trend in the viscosity published by Bencini (2002) can be explained by the use of heat treated cow milk in his experiments.

3.2. CaCl₂ concentration

Our experiments with different $CaCl_2$ concentration (Fig. 3.) resulted similar curves which were showed in Fig 2.

The increase of CaCl₂ concentration resulted shorter cutting time but also the decrease of viscosity. We discovered 44.5 min. cutting time in sample without CaCl₂ adding, but 27.0 min. in the sample adding 1,70mmol CaCl₂, confirming the results of Dalgleish 1983, Castillo 2002, Walstra 2006, Szakály 2001.



Fig. 4. The effect of $CaCl_2$ concentration on the renneting of goat milk (0-250 μ l 10% $CaCl_2$ solution, 0-1,700 mmol)

The cutting time of control sample (without $CaCl_2$) represents a typical soft cheese cutting time, and the $CaCl_2$ added samples had got cutting time like semi-hard cheeses. The decrease of cutting time was strong to adding 50µl $CaCl_2$ solution (5µg/100 ml milk) but adding more $CaCl_2$ solution the scale of cutting time decrease was slower and was equalized in 30 min. approximately (Fig. 5.).



Fig. 5. Correlation of CaCl₂ concentration with the cutting time and the viscosity of curd

Moreover the curd viscosity decreased parallel with the cutting time decrease. The control sample represented the greatest viscosity (632,39 mPas) and the sample adding 250 μ l CaCl₂ solution (25 μ g/100ml milk) showed the weakest curd firmness (viscosity). This finding is contrary with the result of Nájera et al. (2003) but partly agree with the result of Fenyvessy, Csanádi (2007).

Our result contrary with literature can be explained that we use raw goat milk and the use of heat treated goat milk can cause different results. These contrary results need further experiments.

3.3. Temperature

The renneting temperature of raw goat milk was changed from 30° C to 46° C adding 50μ l CaCl₂ solution and 300μ l enzyme solution. The registered renneting curves showed in Fig. 6.



Fig. 6. The effect of temperature on the renneting of raw goat milk (50µl 10% CaCl₂ solution, 300µl 10% enzyme solution)

The temperature affected markedly on the renneting properties of raw goat milk. The lowest temperature (30 °C) resulted the longest cutting time (32.5 min.). The cutting time markedly decreased further to 38°C than seems to equalized around 17 min.. The biggest change was observed between 30 and 34 °C. The enzyme properties as the heat sensitivity or/and temperature of optimum activity can have a role in the equalizing of the cutting time. It also can be possible that the inactivation of enzyme was started at 40 °C (Sholtz 2007).



Fig. 7. Correlation of renneting temperature with the cutting time and curd viscosity

We observed the decrease of viscosity of curd setting higher temperature as we mentioned above. Sample renneted at the lowest temperature $(30^{\circ}C)$ had the greatest viscosity (510.86 mPas), while the sample renneted at the highest temperature (46°C) had the lowest one (80.47mPas) (Fig. 7.). The 16°C temperature difference caused 6.3 fold differences in the viscosity of curd. This extreme difference in the curd viscosity is contrary the results above the cutting time changed greater than viscosity.

Our result is contrary with result of Nájera (2003) related to viscosity affected by temperature. On the other hand, our determined trend in the changing of cutting time affected by temperature agrees with the results of Walstra (2006).

3.4. Predicting of the cutting time

The shape and trend of the observed viscosity curves very similar as published in the literature (Castillo et al. 2000, Castillo et al. 2002). So we used a similar method for working out a cutting time predicting method. We present this method with results originated only from our CaCl₂ addition experiments.

First we used a four degree polynom for description of changes (Fig. 8.).



Fig. 8. The equation of viscosity change

Then we calculate the inflexion point of this polynom and after we investigated the correlation between the time at the inflexion point and the time at the maximum viscosity as cutting time (Fig. 9.).



Fig. 9. Relation between the inflexion point time and the cutting time

We found a linear correlation between the inflexion point time and the cutting time. The close correlation suggests that based on further experiments can work out a common equation what is appropriate to predict the optimal cutting time of rennet curd.

4. CONCLUSION

Investigating the renneting of raw goat milk we observed markedly differences in the cutting time and the curd viscosity. All the increase of temperature, enzyme and $CaCl_2$ concentration caused a decrease in the cutting time and also in the curd viscosity.

Our results related $CaCl_2$ addition was contrary with the literature can explain that we used raw goat milk. The use of heat treated goat milk could give different results partly

which needs further experiments. Based on our results it can be suggest that not the $CaCl_2$ addition is the main goal for the optimization of cutting using raw goat milk for cheese making, because this can cause a markedly decrease in curd viscosity.

Principally, the temperature and the enzyme concentration have to be well determined in the interest of the decrease of losses (curd fines) resulting higher yield and better quality. If we use CaCl₂ addition, using raw milk, only such a CaCl₂ concentration can acceptable, what is not decreases the curd viscosity dramatically, avoided the curd loss.

The effect of the increase of renneting temperature on the cutting time equalized at 38°C but the curd viscosity was markedly lower. Therefore the increase of renneting temperature more than 38°C does not pay making cheese from raw goat milk. More exactly, maximum 34-36°C can be suggested as renneting temperature but very important to emphasize that the use of the optimal enzyme concentration is also principally.

Finally, we suggest that the vibration viscometer is usable to investigation of the gelation of milk, because great differences were observed in the different renneting conditions but further experiments is needed for the determination of the precise cutting time and its correlation with the curve's infelexion point time.

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REFERENCES

- 1. Aleandri, R., Schneider, J.C., Buttazoni L.G. (1989). Evaluation of Milk for Cheese Production Based on Milk Characteristics and Formagraph Measures. Journal of Dairy Science 72, 1967-1975.
- 2. Castillo M., Payne, F.A., Hicks, C.L. Lopez M.B. (2000). Predicting cutting and clotting time of coagulating goat's milk using diffuse reflectance: effect of pH, temperature and enzyme concentration. International Dairy Journal 10, 551-562.
- 3. Castillo, M., Payne, F.A., Hicks, C.L., Laencina, J., Lopez, M.B. (2002). Effect of calcium and enzyme in cutting time prediction of coagulating goats' milk using a light scattering sensor Inernational Dairy Journal, 12. 1019-1023.
- 4. Dybowska, B.E., Fujio, Y. (1996a). Effect of temperature and glucono-d-lactone (GDL) concentration on milk aggregation and gelation process as revealed by optical method. Milchwissenschaft, 51(10), 557-560.
- 5. Dybowska, B.E., Fujio, Y. (1996b). Effect of temperature and glucono-d-lactone (GDL) concentration on milk aggregation and gelation process as revealed by optical method. Milchwissenschaft, 51(10), 557}560.
- 6. Eleya, M.M.O., Banon, S.D., Hardy, J. (1995). A comparative study of pH and temperature effects on the acidic coagulation of milks from cows, goats, and sheep. Journal of Dairy Science, 78, 2675-2682.
- 7. Fenyvessy, J., Csanádi, J. (2007): Dairy technology (Tejipari technológia, Jegyzet Universitas Kiadó) University Publishing Szeged p. 202-204.
- 8. Guthy, K., Novak, G. (1977). Observations on the primary phase of milk coagulation by rennet under standardized conditions. Journal of Dairy Research, 44, 363-366.
- 9. Hardy, J., Fanni, J. (1981). Application of reflection photometry to the measurement of milk coagulation. Journal of Food Science, 46, 1956-1957.
- 10. Hori, T. (1985). Objective measurements of the process of curd formation during rennet treatment of milk by the Hot Wire method. Journal of Food Science, 50 (4), 911-917.

- 11. Korolczuk, J., Maubois, J.L., Loheac, J. (1986). Suivi de la coagulation- preH sure du lait a` l'aide d'un nouveau capteur refractometrique. Le Lait, 66 (4), 327-339.
- 12. Lochte-Watson, K.R., Payne, F.A., Gates, R.S., Hicks, C.L. (1998). Effects of pH, protein, fat, and calcium on diffuse reflectance of milk. Transactions of the ASAE, 41(3), 701-707.
- 13. McMahon, D.J., Brown, R.J. (1982). Evaluation of Formagraph for comparing rennet solutions. Journal of Dairy Science, 65, 1639-1642.
- 14. McMahon, D.J., Brown, R.J., Ernstrom, C.A. (1984). Enzymic coagulation of milk casein micelles. Journal of Dairy Science, 67, 745-748.
- 15. McMahon, D. J., & Brown, R. J. (1990). Development of surface functionality of casein particles as the controlling parameter of enzymic milk coagulation. Colloids and Surfaces, 44, 263-279.
- 16. Payne, F.A., Madangopal, S., Hicks, C.L., Shearer, S.A. (1990). Fiber optic milk coagulation sensor for cut-time detection. Proceedings of the Food Processing Automation Conference AEAE, St. Joseph, MI. In: Castillo M., Payne, F.A., Hicks, C.L. Lopez M.B. (2000). Predicting cutting and clotting time of coagulating goat's milk using diffuse reflectance: effect of pH, temperature and enzyme concentration. International Dairy Journal 10, 551-562
- 17. Payne, F.A., Hicks, C.L., Shen, P.S. (1993a). Predicting optimal cutting time of coagulating milk using diffuse reflectance. Journal of Dairy Science, 76, 48-61.
- 18. Payne, F.A., Hicks, C.L., Madangopal, S., Shearer, S.A. (1993b). Fiber optic sensor for predicting the cutting time of coagulating milk for cheese production. Transactions of the ASAE, 36(3), 841-847.
- 19. Richardson, G.H., Okigbo, L.M., Thorpe, J.D. (1985). Instrument for measuring milk coagulation in the cheese vats. Journal of Dairy Science, 68, 32-36.
- 20. Scher, J., Hardy, J. (1993). Study of the evolution of casein micelle size distribution after renneting by means of quasielastic light scattering. The Australian Journal of Dairy Technology, 48, 62-65.
- Szalai B. (2008): Investigation of renneting of cow milk. Thesis. USZ Faculty of Engineering (Tehéntej oltós alvadásának vizsgálata. Szakdolgozat SZTE-MK), Szeged
- 22. Ustunol, Z., Hicks, C.L. (1990). Effect of a coagulation monitoring device on experimental cheese yield. Journal of Dairy Science, 73, 1-7.