Online Version ISSN: 1314-412X
Volume 4, Number 4
December 2012

AGRICULTURAL SCIENCE AND TECHNOLOGY

2012

An International Journal Published by Faculty of Agriculture,
Trakia University, Stara Zagora, Bulgaria

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Monitoring of milk acid coagulation by rotational viscometer

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Abstract. *In the following work possibilities to monitor the acid coagulation dynamics by rotational viscometer are discussed. There is established suitable speed of rotation of the cylinder, in line with the delicate structure of the gel. The time to achieve the isoionic point in the acid coagulation of milk can be determined by changing the viscosity. The obtained results give grounds to assert that the viscometers cannot be used alone for the detailed monitoring of acid coagulation. Viscometers can be used as the reference method for monitoring changes of dynamic viscosity of milk during acid coagulation and determining the point of gelation. To establish the parameters of acid coagulation dynamics titratable acidity (°T), pH and lactic acid accumulation on milk (%) were followed.*

Keywords: acid coagulation, viscometers, milk

Abbreviations: TA – titratable acidity, LA – lactic acid

Introduction

Acid coagulation is the critical first step in the manufacturing of yoghurt products. This process of acid formation has a significant influence on texture, microstructure and rheology of yogurt, contributing to the overall sensory and visual perception by the consumer. Acid gels may be considered as a protein network composed of demineralized casein micelles formed as a result of gradual fermentation of lactose by lactic acid bacteria (Horne, 1999). As a result of gradual accumulation of lactic acid, respectively lower pH, physical and chemical changes in micellar structure of casein are observed. These changes are especially significant at pH 5.3 ÷ 5.0 where the basic structural element in casein micelles colloidal calcium phosphate (CCP) is dissolved completely in the liquid phase (Dalgleish, 1989; Gastaldi, 1996). As a consequence of these processes changes in rheological characteristics of milk (viscosity, elasticity, etc.) are established due to destabilization and transformation of micelles which phosphorous-calcium bounds are destroyed.

The rheological properties of milk during acid coagulation can be assessed by a range of rheological techniques such as small amplitude oscillatory rheometers, large amplitude oscillatory shear, penetometers, rotational viscosimeters (Benezech and Maingonnat, 1994; Velez-Ruiz and Barbosa Canovas, 1997). Combination between rheological techniques and other instrumental methods may be used to monitor the gel formation phase, as well as the impact of subsequent processing steps on the properties of the gel.

The objective of this study is to establish a possibility for monitoring the structure formation during acid coagulation of milk by using different speeds of rotation of the cylinder of rotational viscometer.

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Material and methods

Preparation of yogurt

In this study low-fat cow's milk 1.5% is used. The pasteurized milk bases is cooled to incubation temperature 45°C, inoculated with lactic starter (*Lactobacillus delbrueckii ssp.bulgaricus u Streptococcus thermophilus*), poured into 400 ml plastic containers and incubated at 45°C in the conditions of water bath. With the information and archiving system Microsyst – MS Data Logger Network 3.02 data are recorded in real time to reach pH 4.6 (the pH end point) and T (°C) (Figure 1). Titratable acidity (represented as % lactic acid) and dynamic viscosity (Pa.s) determination were conducted at fixed time intervals during the incubation of yogurt.

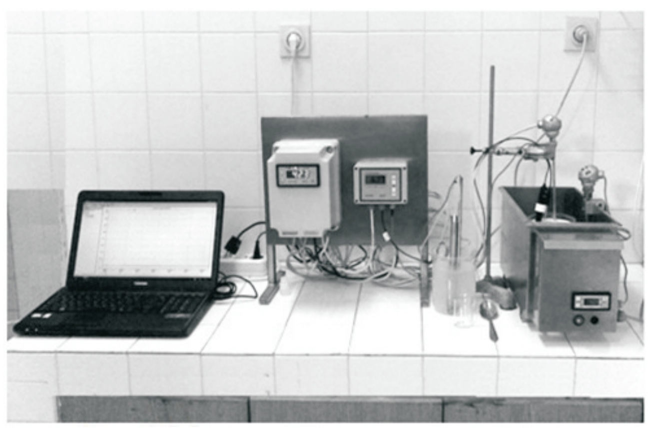

Figure 1. Laboratory system for control and archiving.

Determination of titratable acidity

Titratable acidity is determined at intervals of 20 min by double titration of milk samples of 10 ml with 0.1 n NaOH using phenolphthalein as indicator. Results are represented as lactic acid (%) Using the current value for TA (TA_n) and initial (TA_0) the amount of lactic acid (LA,%) (CH3-CHOH-COOH, MW = 90) is obtained using the following formula:

$LA = (\Delta TA \times MW \times n) / 1000, \%$

where:

LA – lactic acid, (%);

ΔTA – variation of titratable acidity, (°T);

MW – molecular weight of lactic acid, Da;

0.009 – conversion coefficient of Thorner degrees in lactic acid;

n – normality of NaOH.

Variation of TA (ΔTA) is obtained as the difference between the current value (TA_n) and initial value (TA_0) measured after the addition of starter culture (TK_o).

$\Delta TA = TA_n - TA_{o,}$ °T

For n = 0.1 is obtained:

$LA = \Delta TA \times 0.009, \%$

Measurement of viscosity

Dynamic viscosity (Pa.s) is measured using a rotational viscometer model Reotest 2.1, (VEB MLW Prüfgeräte-Werk Medingen, Germany) with measuring cylinder type N, which is used to measure the viscosity of light liquids. In dealing with this kind of measuring cylinder the required amount of sample is 11 ml, the samples examined must show a viscosity in the range of 1 ÷ 200 000 mPa.s, applied shear stress (τ_r) is in the range of 1,6 ÷ 32 Pa and velocity shear (Dr) from 0.15 to 1310 s^{-1}. At the measuring tube 11 ml of sour milk were placed. Viscosity measurements are performed in sour milk at the incubation temperature 45°C, which is maintained by a circulating water bath. Every 20 min, it is taking the values of α = (0 ... 100) which are recorded from the indicator device. Viscosity measurements are made at four speeds of cylinder shown in Table 1.

Table 1. Rotation speeds of the cylinder type N.

Measuring cylinder	Step of speed (rate of gear)	Rotation speed min^{-1}
N	1 bd	0.28
	2 bd	0.50
	3 bd	0.83
	4 bd	1.50

For each speed dynamic viscosity is calculated by the following equation:

$\eta = (\tau_r / D_r) \times 100$, (Pa.s)

where:

D_r – shear rate, (s^{-1});

τ_r - shear stress, (10^{-1} Pa).

Shear stress is calculated by the formula:

$\tau_r = z \times \alpha$, (10^{-1} Pa)

where:

z - constant of the cylinder, (10^{-1} Pa). Constant of cylinder type N has a value of 3.44(10^{-1} Pa);

α – recorded value of the indicator device (0...100);

Results and discussion

Using spline-function of the program SigmaPlot (Version 12.0) was carried out connecting the intermediate values to those obtained in the experiment. The accumulation of lactic acid during acid coagulation (Figure 2) is realized by about 0.05 % to 20 min and for the period of 160 min its amount reached 0.42 % lactic acid. The resulting coefficient of correlation R = 0.0026 obtained by linear regression equation of the type $y = y_o + ax$ at significance level $P < 0.0001$ showing the linear character of the process.

Using the rotation speed of the measuring cylinder 0.28 min^{-1}

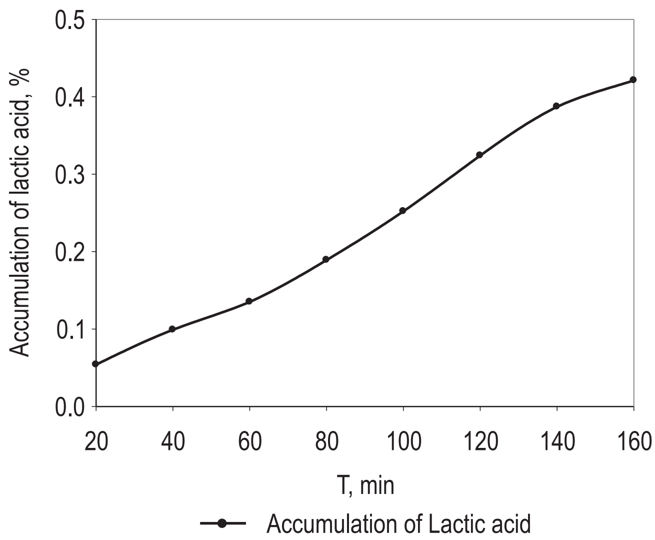

Figure 2. Accumulation of lactic acid (%) during lactic acid fermentation.

(Figure 3) initial changes in the viscosity of milk at pH = 5.5 are established. These changes are not accompanied by the visible coagulation typical for pH = 5.3 ÷ 5.1. After achieving pH = 5. 4 no change is observed in the values of the viscosity due to the formation of protein aggregates on the surface of the cylinder, leading to blockage of his movement. Initial changes in viscosity cannot be established by the higher speeds of rotation of the cylinder (0.50, 0.83 and 1.50 min^{-1}) (Figure 4, 5, 6). The higher speed gives the possibility to establish the time of initial coagulation at pH = 5.3 and inability to follow the end of the coagulation at pH = 4.6 due to their destructive effect on the gel formations.

At a speed of rotation of the cylinder 0.50 min^{-1} after reaching pH = 5.1 a phase without change in viscosity is established due to the presence of the above-mentioned reason. At a speed of 0, 83 min^{-1}, in order to obtain particles with a size comparable to the distance between the working cylinders, we can properly monitor the process of acid coagulation. Due to the higher speed compared to the speed, the study of which is shown in Figures 3 and 4, aggregation particles that reach the maximum size get destroyed, those of which the size is comparable to the distance between the working cylinder and the recipient, slowing rotation of the rheometer cylinder body, their destruction leads to uneven progress of received data for the dynamic viscosity.

At a speed of 1,50 min^{-1}, after reaching pH = 5.1 the gel formed is destroyed, but because of the higher speed particles are dispersed evenly. This is the reason for dynamic viscosity to remain at pH = 4,7 ÷ 4,8, then the spinning cylinder moving with greater speed times dispersed coagulum to smaller particle size, as shown by a decrease in the values of dynamic viscosity.The maximum value for dynamic viscosity is reduced by increasing the speed of rotation of the working cylinder 23000.10^{-4} Pa.s to 4300.10^{-4} Pa.s. This means that the destruction of the acid gel at a speed 1,50 min^{-1} began at considerably earlier stage of the slower speeds.

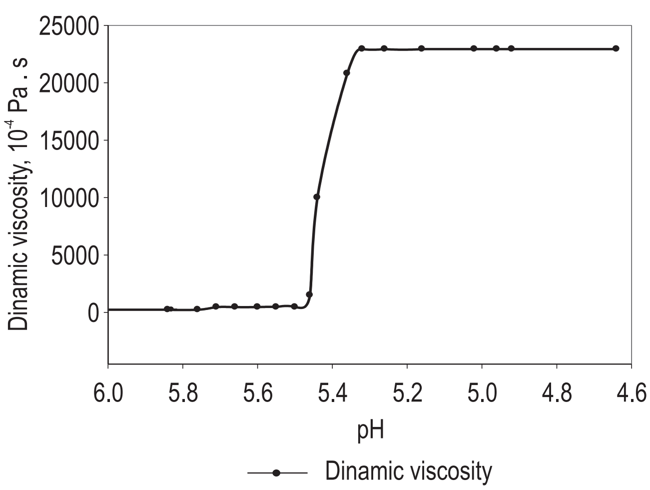

Figure 3. Change of viscosity with rotational speed of the cylinder 0.28 min^{-1}.

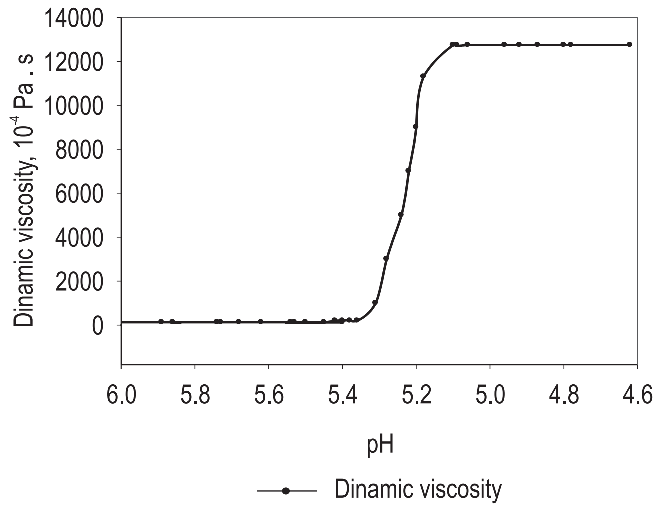

Figure 4. Change of viscosity with rotational speed of the cylinder 0.50 min^{-1}.

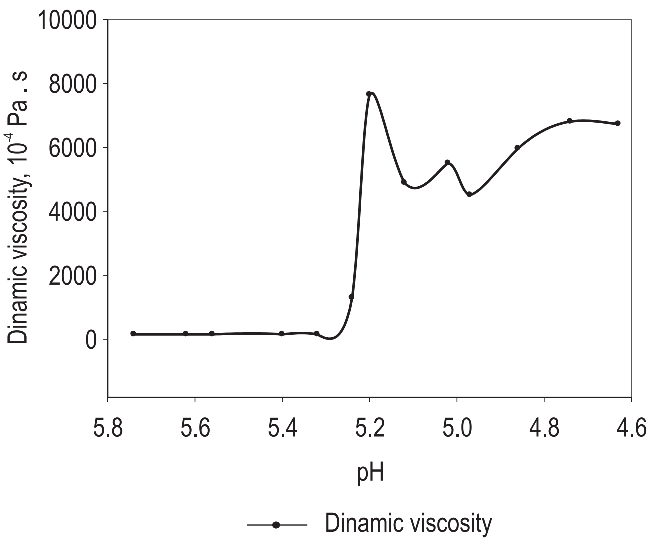

Figure 5. Change of viscosity with rotational speed of the cylinder 0.83 min^{-1}.

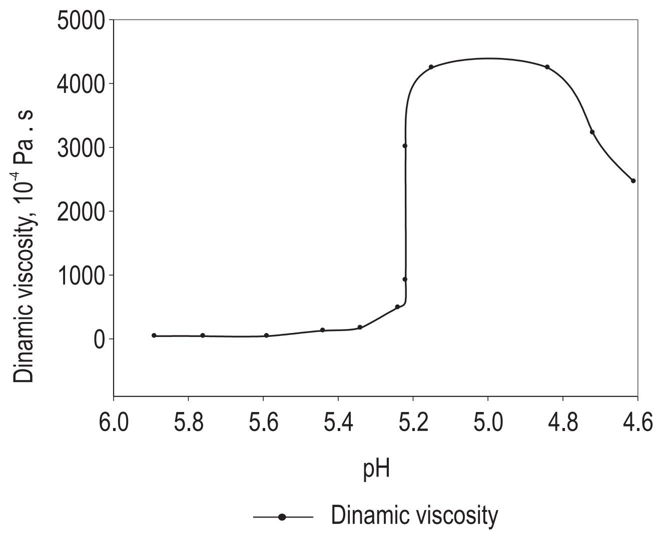

Figure 6. Change of viscosity with rotational speed of the cylinder 1.50 min^{-1}.

Conclusion

Application of low speed 0.28 min^{-1} cylinder is suitable for establishing the initial changes in structure formation during acid coagulation of milk but is not suitable for capturing the end time of gel formation. Use of higher speeds (0.50; 0.83 and 1.50 min^{-1}) gives adequate indication of the formation of aggregates but as a result of their destructive effect on the gel, the process of coagulation has to be monitored.

Future research will follow the acid coagulation using a combination between speed of rotation of the measuring cylinder. The aim of shifting into another gear when it reaches a certain pH is to ignore the influence of protein aggregates resulting from acid coagulation.

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AGRICULTURAL
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Volume 4, Number 4
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