A C T A A L I M E N T A R I A P O L O N I C A
Vol. XIV (XXXVIII), No. 2
1988

WACŁAW LESZCZYŃSKI
ANTONI GOLACHOWSKI

COMPARISON OF POTATO STARCH GELATINIZATION TEMPERATURES DETERMINED BY DIFFERENT METHODS

Department of Food Storage and Technology, Agricultural University, Wrocław

Key words: potato starch, gelatinization temperatures.

The temperatures of potato starch gelatinization were compared using four methods of determinations, i.e. Brabender's viscograph, rotary viscosimeter, microscopic and conducting methods. The results of gelatinization temperatures determined by the methods mentioned above were different. They showed correlation in each case excluding when the results of the final temperature were obtained using Brabender's viscograph.

INTRODUCTION

Very important data characterising starch properties are the temperatures observed at the beginning and at the end of starch pasting. There is a lot of methods used to determine the gelatinization temperatures. They are based on the observations of changes in the properties of starch granules and suspensions which occur when starch is heated with water. A number of these methods cannot be considered as objective and apart from that they are labour — and time — consuming. Recently, some new methods based on the differences in the amount of heat taken by the pasting starch suspension [12], changes in the starch X-ray picture [13] or rising susceptibility of the pasting starch to enzymatic decomposition [2] have been elaborated. However, the methods mentioned above require the use of very expensive equipment or very high precision from the operators. For this reason they cannot be widely used in industrial laboratories.

The microscopic methods belong to the most common methods used in the determinations of gelatinization temperatures. They are based on the changes in the microscopic picture of the granules, especially in polarized light [11]. Another method frequently used is a viscosimetric method based on the changes in viscosity of water starch suspensions during heating [5]. One of the viscosimetric methods is that based on the use of Brabender's viscograph [5], which gives reproducible results. Its disadvantage is that the value of temperature being the function of time in the determination can be read on the recorder tape. The temperatures of starch pasting by viscosimetric method can be determined also by means of a rotary viscosimeter [6]. It does not cause any of the problems mentioned above when it is connected to the recorder in temperature — viscosity relation. However, the results obtained on a viscosimeter depend on the angular velocity of the stirrer and to a great extent on the concentration of starch suspensions used in the experiment [6]. The disadvantage of all viscosimetric methods is high dependence of the results on the type of the equipment and on the conditions under which the determinations are carried out [3,4,10].

Since viscosimetric methods are not found to be satisfactory, some efforts have been made to elaborate new, objective and simple methods which could be used in the determinations of starch gelatinization temperatures. One of them is an objective microscopic method based on the observations of birefringence decay of starch granules in polarized light. A subjective visual reading in this method has been replaced by automatic recording of the changes in intensity of the light emitted by the granules in polarized light under the microscope. These changes were transmitted to the recorder in X-Y system by means of a sensitive photoelement [7]. Another method elaborated for this purpose is the determination of starch gelatinization temperatures on the basis of automatic recording of the changes in the intensity of electric current being conducted by water starch suspension during heating. The results of the determinations obtained by this method did not depend on starch concentration in the suspension [7].

The purpose of the study was to compare the results of the determinations of potato starch gelatinization temperatures obtained by four simple and objective methods: Brabender's viscograph, rotary viscosimeter, microscopic and conducting methods.

MATERIAL AND METHODS

The material used in the experiment included 23 samples of the potato starch obtained in various starch plants in the years 1976-1984.

Four methods were used to determine the starch gelatinization temperatures (initial and final), i.e.:

1. Viscosimetric method using Brabender's viscograph. The determinations were made in 5% water suspension of starch. The suspension was heated at the rate of 1,5°C/min starting from 40°C up to 96°C. The recorder tape feed was 0,5 cm/min. The accuracy of temp. reading was 0,5°C. The time of determination for 1 sample in two replications reached approximately 120 minutes.

2. Viscosimetric method using a rotary viscosimeter "Rotovisko RV-3" of Haake with programmer PG-11 and FT ultrathermostat. The determinations were carried out using 7% water suspension of starch. The measuring element was a stirrer with a double wing, the rotations of which were 600 r.p.m. The suspension was heated at the rate of 2°C/min starting from 45°C up to 80°C. The accuracy of temperature reading was 0,1°C. The time of the determination for 1 sample in two replications was about 80 minutes.

3. Objective microscopic method. The microscope used in the experiment was provided with a heated table Boetius and polarizing filters. The apparatus was connected to a photoelectric device [8] and a recorder PM 8120 in XY system. The water suspension of starch was placed on the table of the microscope and heated at the rate of 7°C/min starting from 35°C up to 80°C. The accuracy of temperature reading was 0,25°C. The time of the determination for 1 sample in five replications was about 60 minutes.

4. Conducting method. The rotary viscosimeter "Rotovisko RV-3" was adjusted with a device transmitting the changes in conductions of the pasting starch suspension to the recorder PM 8120 [9]. 7% water suspension of starch used in the experiment was heated at the rate of 2°C/min starting from 45°C up to 80°C. The accuracy of temperature reading was 0,25°C. The time of the determination for 1 sample in two replications reached about 80 minutes.

Figure shows the method of determination of initial and final gelatinization temperatures of starch.

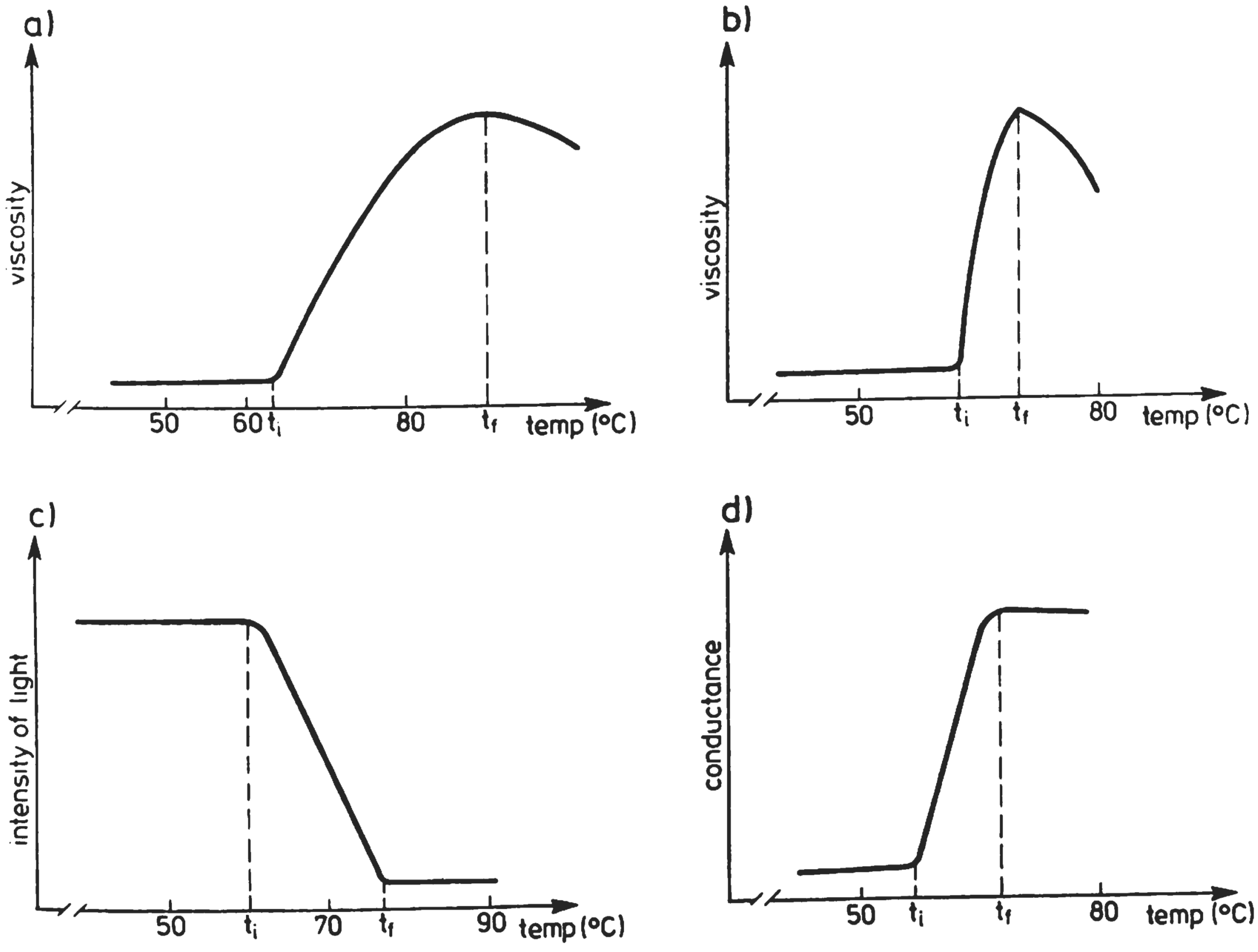

Fig. Methods of determination of gelatinization temperatures of starch; a — Brabender viscograph, b — rotary viscosimeter, (Rotowvisko — RV-3), c — microscopic method, d — conducting method, t_i — initial gelatinization temperature, t_f — final gelatinization temperature

The results obtained in the experiment were calculated statistically. The significant difference between the mean values was determined on the basis of the criterion of the least significant difference (LSD). Moreover, correlation coefficients were calculated.

RESULTS

Table 1 shows the mean values and the range of starch gelatinization temperatures. The initial temperatures of starch gelatinization determined by the four methods described above varied significantly. The highest values of initial gelatinization temperature were observed when Brabender's viscograph was used for the measurements (64,1°C on average). The initial gelatinization temperatures were lover when they were determined on a rotary viscosimeter (63,7°C) and by microscopic method (60,0°C). The lowest values were observed when the conducting method was used (57,3°C). The initial gelatinization temperatures were found within the range from 3,5°C (62,5-66,0) to 4,3°C (57,6-61,9).

The results of the final gelatinization temperature determined on a rotary viscosimeter and by conducting method did not show any significant differences and they were found to be the lowest among all the four values being determined (Tab. 1). When the microscopic method was used, the temperature was approximately 6°C higher than in other cases. However, the highest values of final temperature of gelatinization (90°C on average) were obtained when Brabender's viscograph was used. The highest temperature span, i.e. 16,5°C (79,5-96) was observed when other methods were used the temperatures were found within the range from 3,0°C (73,5-76,5)—microscopic method to 4,5°C (66,3-70,8)—conducting method.

Table 1. Potato starch gelatinization temperatures determined by 4 methods

Temperature (°C)		Methods				LSD, α=0,05
		Brabender's viscograph	rotary viscosimeter	microscopic	conducting	
Initial (t_i)	from—to	62.5—66.0	62.0—65.8	57.6—61.9	55.6—59.3	—
	mean	64.1	63.7	60.0	57.3	0.2
Final (t_f)	from—to	79.5—96.0	67.0—70.9	73.5—76.5	66.3—70.8	—
	mean	90.5	68.3	74.8	68.7	1.2
Range (t_f-t_i)		26.4	5.0	14.8	11.4	—

Range of temperature gelatinization (the differences between the initial and final gelatinization temperatures) for the starch samples used in the experiment using four various methods were as follows: 26,4°C using Brabender's viscograph, 14,8°C for microscopic method, 11,4°C for conducting method and 5,0°C when a rotary viscosimeter was used.

Table 2 shows the correlation coefficients obtained for four methods used in the experiment. The initial temperatures of starch gelatinization obtained by means of Brabender's viscograph, rotary viscosimeter, microscopic and conducting method were correlated with one another and the correlation coefficients ranged from 0,926 to 0,990.

Table 2. Corrlelation coefficient between the results of the potato starch gelatinization temperatures determined by 4 methods

Method		Brabender's viscograph	Rotary viscosimetr	Microscopic	Conducting
		final temperatures			
Brabender's viscograph	initial temperatures		0.296	0.297	0.029
Rotary viscosimetr		0.976*		0.728*	0.770*
Microscopic		0.976*	0.948*		0.739*
Conducting		0.960*	0.965*	0.990*	

* Singnificant correlation

On the other hand, the results of final temperatures of starch gelatinization obtained using Brabender's viscograph were not correlated with the results of other methods used in the experiment. The results of final temperatures of starch gelatinization determined by means of a rotary viscosimeter, microscopic and conducting method were correlated with one another and the values of correlation coefficients ranged from 0,728 to 0,770.

DISCUSSION

The data in Table 1 show that initial temperatures of starch gelatinization determined by viscosimeter methods (using Brabender's viscograph and rotary viscosimeter) were considerably higher than those determined by microscopic and conducting methods. The viscosimetric methods require large amounts of starch granules to become pasted so that the viscosity of the solution reaches such a level that can be recorded by the apparatus. On the other hand, the photoelement in the microscopic method records the changes in the light intensity resulting from the decay of birefringence of several starch granules (even large ones) which are the first to undergo the pasting process. The initial temperature of starch gelatinization determined by the conducting method was about 3°C lower than that in the microscopic method (tab. 1). Probably a part of carbohydrate substance and the ions bound with it are transmitted to the solution thus resulting in the changes of its conductivity at lower temperatures than birefringence decay is observed. Even slight changes in the current conductivity were recorded by the measuring device.

Starch gelatinization of the samples determined by the conducting method was finished at 66,3-70,8°C. Presumably, all the ions present in the pasting granules, were transported to the solution. At such temperatures, a part of small granules can still exhibit birenfringence, therefore, the final temperature of starch gelatinization determined by the microscopic method was higher and ranged from 73,5 to 76,5°C.

The difference between the final temperature of starch gelatinization determined using Brabender's viscograph and a rotary viscosimeter was likely due to the use of varied concentration of the starch suspension. The differences in the final temperature of starch gelatinization could also be affected by the stirrer placed in the rotary viscosimeter reaching the rate of 600 revolutions/min, subsequently disintegrating the structure of the paste, thus, reducing the recorded viscosity before all the granules have become totally pasted. This can also be due to the smallest difference between the initial and final temperature of starch gelatinization (5°C) among all the methods used in the experiment.

The differences between the gelatinization temperatures determined by various methods have also been proved by other authors [1].

The least time consuming method among those used in the determinations was the microscopic method. It was characterized by great accuracy of temperature reading, i.e. 0,25°C. When large portions of starch are examined, the results obtained in this way can by no means be representative since very small quantities of starch are used in the determination, i.e. only a few miligrams.

The determinations of starch gelatinization temperatures on a rotary viscosimeter were characterized by the reading accuracy of 0,1°C. The use of the high speed stirrer in the rotary viscosimeter probably affected the structure of the paste which could be subsequently reflected in the results of the determinations.

The time used in the conducting method was the same as that used in case of the rotary viscosimeter, and the reading accuracy was the same as in microscopic method. Similarly as in viscosimetric methods, larger starch samples used in the determinations can be representative.

The method using Brabender's viscograph turned out to be the most time-consuming of all the methods and the reading accuracy was 0,5°C being the lowest among those used in the experiment. The lack of correlation between the results of final temperature of starch gelatinization determined by this method and by the three other methods can account for the fact that Brabender's viscograph is not bery useful in this particular case.

The conducting and microscopic methods can be used in the determinations of gelatinization temperatures which has been proved by high correlation coefficient between the results obtained using these methods and viscosimetric methods as well as their accuracy and the fact that they are not very time consuming. The conducting method is more suitable when larger portions of starch are examined, whereas microscopic method is better for smaller starch portions taken for analysis.

CONCLUSIONS

The following conclusions can be drawn from the results obtained in the experiment:

1. The temperatures of potato starch gelatinization determined by means

of Brabender's viscograph, rotary viscosimeter, microscopic and conducting methods were different.

2. The highest values of initial and final temperatures of gelatinization were obtained when Brabender's viscograph was used and the lowest values were obtained for the conducting method.

3. The results of the initial temperatures of gelatinization determined by all four methods were highly correlated.

4. The results of final temperature of gelatinization determined by Brabender's viscograph were not correlated with the results obtained using other methods.

LITERATURE

1. Goehring K. J., Fritts D. H., Allen K. G. D.: Cereal Chem., 1974, **51**, 764.
2. Goshima G., Ohashi K., Tsuge H., Ohno Y.: Res. Bull. Fac. Agr. Gifu Univ., 1978, **41**, 115.
3. Goto F., Yokoo Y.: Starch/Stärke 1969, **21**, 128.
4. Juliano B. O., Peres C. M., Alyoshin E. P. et al.: Starch/Stärke 1985, **37**, 40.
5. Kempf W.: Starch/Stärke 1955, **7**, 161.
6. Koubek I.: Starch/Stärke 1974, **26**, 81.
7. Leszczyński W.: Starch/Stärke 1987, **39**, (in press).
8. Leszczyński W., Mucha Z.: Patent PRL 125859 (1.09.1982).
9. Leszczyński W., Mucha Z.: Patent PRL 137920 (27.12.1985).
10. Oosten B. J.: Starch/Stärke 1980, **32**, 272.
11. Schierbaum F., Taüfel K.: Starch/Stärke 1962, **14**, 411.
12. Stevens D. J., Elton G. A. H.: Starch/Stärke 1971, **23**, 8.
13. Takeda Ch., Hizukuri S.: J. Agr. Chem. Soc. Jap., 1974, **48**, 663.

Manuscript received: February, 1987
Authors address: 50-375 Wrocław, Norwida 25.

W. Leszczyński, A. Golachowski

PORÓWNANIE TEMPERATURY KLEIKOWANIA SKROBI ZIEMNIACZANEJ OZNACZONEJ RÓŻNYMI METODAMI

Katedra Technologii Rolnej i Przechowalnictwa, Akademia Rolnicza Wrocław

Streszczenie

Jest wiele metod oznaczania temperatury kleikowania skrobi opartych na obserwacji zmian różnych właściwości gałeczek i zawiesin skrobi zachodzących podczas ogrzewania z wodą. Niektóre metody wymagają stosowania kosztownej aparatury, inne są pracochłonne lub nieobiektywne, co uniemożliwia stosowanie ich w laboratoriach przemysłowych.

Celem pracy było porównanie wyników oznaczeń temperatury kleikowania skrobi ziemniaczanej wykonanych czterema prostymi, zobiektywizowanymi metodami. Zastosowano dwie metody wiskozymetryczne — przy użyciu wiskografu Brabendera i wiskozymetru rotacyjnego „Rotovisko RV-3" oraz dwie nowo opracowane metody: mikroskopową i przewodnościową.

Zobiektywizowana metoda mikroskopowa polega na automatycznym rejestrowaniu zaniku dwułomności gałeczek podczas ogrzewania na podstawie zmian natężenia światła spolaryzowanego,

przechodzącego przez gałeczki, a metoda przewodnościowa oparta jest na automatycznym rejestrowaniu zmian przewodnictwa elektrycznego zawiesiny skrobi podczas ogrzewania.

Na rys. 1 przedstawiono przyjęte sposoby wyznaczania temperatury kleikowania skrobi.

Na podstawie obliczeń statystycznych stwierdzono, że wyniki uzyskane stosowanymi metodami różniły się między sobą (tab. 1). Najwyższe wartości temperatury początkowej i końcowej kleikowania otrzymano przy użyciu wiskografu Brabendera (temp. początkowa średnio 64,1°C, temp. końcowa — 90,5°C), a najniższe przy zastosowaniu metody przewodnościowej (temp. początkowa 57,3°C, temp. końcowa 68,7°C). Wyniki oznaczeń temperatury początkowej kleikowania oznaczone stosowanymi metodami były ze sobą skorelowane, a wartości współczynników korelacji wahały się od 0,926 do 0,990. Natomiast wyniki oznaczeń temperatury końcowej kleikowania wykonane przy zastosowaniu wiskografu Brabendera nie były skorelowane z wynikami otrzymanymi przy użyciu pozostałych metod (tab. 2).

Opracowane metody — przewodnościowa i mikroscopowa — mogą być stosowane do oznaczeń temperatury kleikowania. Przemawiają za tym wysokie współczynniki korelacji między wynikami otrzymanymi tymi metodami, a metodami wiskozometrycznymi oraz ich dokładność i stosunkowo mała czasochłonność.

Metoda przewodnościowa jest bardziej odpowiednia w przypadku badania większych partii skrobi, a mikroskopowa zwłaszcza wówczas, gdy dysponuje się małymi ilościami materiału do analiz.