

Starch Gelatinization in Pasta Cooking: Differential Flux Calorimetry Investigations¹

M. RIVA, L. PIAZZA, and A. SCHIRALDI²

ABSTRACT

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Pasta cooking conditions were simulated in a differential flux calorimeter. The profile of the endothermic starch gelatinization peak was observed to directly describe the process according to first-order kinetics. An early small exothermic effect was attributed to wetting. Isothermal calorimetry provides much more reliable results than do other traditional

approaches, and it allows comparison among different kinds of pasta. This work gives a detailed presentation of the physical meaning underlying the calorimetric trace, which allows assessment of the gelatinization kinetics and simple thermodynamic treatment.

Starch gelatinization is of utmost importance in a number of food modifications, such as those that take place in cooking, baking, drying, and extruding starch-based foods. The overall starch gelatinization process is generally supposed to obey a first-order kinetics and depends on temperature (Lund 1989). Nonetheless, the phenomenon is very complex (Atwell et al 1988) and implies significant changes in physical, chemical, and nutritional properties of the starch as well as water (Aguerre et al 1989, Levine and Slade 1989) and heat diffusivity, viscosity, rheological

behavior, swelling and deformation of the original shape of starchy products, and susceptibility to enzymatic digestion.

The presence of gluten as a further component of the system can modify the availability of water to the starch with reduction of both granule swelling and starch leaching, and improves final quality of the starchy products (Ghiasi et al 1983, Hansen and Jones 1977). Starch gelatinization has been studied through several experimental approaches, including the following:

1. Traditional methods such as polarized light microscopy (Grzybowski and Donnelly 1977), swelling and solubility measurements (Leach et al 1959, Bagley and Christianson 1982), amylose-iodine blue value method (Wotton and Chaudhry 1980), and enzymatic assay (Shetty et al 1974).

2. Viscosity measurements such as viscoamylographic methods, to support rheological models for the mechanical properties in the course of the gelatinization process (Rasper 1976).

3. Thermomechanical and dynamicomechanical analysis, to single out the contributions of starch and gluten to the overall

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²DISTAM, Sez. Tecnologie Alimentari, Universita' di Milano, Via Celoria, 2, 20133 Milano, Italy.

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rheological properties (Masi et al 1990; Cavella et al, in press).

4. More refined microscopic investigations such as scanning electron microscopy and staining techniques, mainly to study the ultrastructure of extruded products (Resmini and Pagani 1983).

5. X-ray diffraction and nuclear magnetic resonance, to assess changes in crystallinity within starch granules (Varriano-Marston et al 1980, Owus-Ansah et al 1982).

6. Differential scanning calorimetry (DSC), to describe the phase transitions in the water-starch systems with a quantitative evaluation of the relevant enthalpy changes (Lund 1984).

Some of these methods give a qualitative description of the process, while others allow a more quantitative interpretation. The method used can affect the conclusions of the investigation (Lund 1984). For example, the values of the kinetic parameters obtained from enzymatic and chemical (iodine-amylose blue value method) approaches are not comparable with those drawn from DSC investigations, while in the very popular case of pasta cooking, assessment of proper cooking time is more accurate when approached via rheological or optical (loss of birefringence) methods (Piazza et al 1990a). On the other hand, a more quantitative approach to the gelatinization kinetics, allowed by DSC determinations, yields results suitable to model descriptions of the microscopic events that occur in the same process (Burt and Russel 1983, Wirakartakusumah 1981). At present, DSC investigations are supposed to be more adequate to study starch gelatinization and retrogradation (staling) (Atwell et al 1988). However, the results in the literature mainly concern nonisothermal DSC investigations and were obtained from small mass samples of native, rice, potato, and maize starch (Biliaderis et al 1980). No DSC investigations have reported on more complex starch formulations, such as pasta products, or on large mass samples.

The present work concerns isothermal investigations carried out with a differential flux calorimeter that allows the study of large mass samples in conditions very close to actual pasta cooking (100°C), that is, with a starch-water ratio of 0.1. The aim of this work was to assess in a quantitative way the kinetic parameters of the starch gelatinization in order to characterize the different behaviors of various types of pasta. A further goal was to quantitatively define the thermodynamics of the process.

MATERIALS AND METHODS

Materials

The following kinds of spaghetti from industrial production lines were used: durum wheat commercial pasta dried at 90°C for 5 hr (durum-HT, water 11.4%, total protein 15.96%), diameter 1.8-mm, manufacturer-recommended cooking time 10–11 min; soft wheat experimental pasta dried at 55°C for 15 hr (soft-LT, water 10.56%, total protein 13.79%), diameter 1.8 mm; and soft wheat experimental pasta dried at 90°C for 5 hr (soft-HT, water 10.55%, total protein 13.50%), diameter 1.8 mm. The operating conditions for the industrial preparation of these products are reported elsewhere (Piazza et al 1990b). Durum HT spaghetti was used either in the original shape or finely ground in a Waring Blender.

Methods

Calorimetric investigations. We used a differential flux calorimeter (C80, Setaram, Lyon, France) that hosts two twin cylindrical inox steel cells with peculiarities (pressure resistance, mixing facilities, controlled gas perfusion, etc.) and can be designed according to the scope of the investigation. The large capacity (10–15 ml) of the cells allows the study of specimens with a mass that ensures reliable sampling of real systems.

The temperature of an isothermal investigation was preset, and thermal equilibrium throughout the calorimeter was attained before recording started (about 1 hr after the introduction of the cells). Detection sensibility also was preset according to the expected intensity of the thermal effect. It can be tuned as the full-scale output voltage, which in turn corresponds to the detectable thermal power, dQ/dt ; the smallest reliable signals are on the order of 1 mJ.

To simulate the actual cooking conditions, a special calorimetric cell was used (Fig. 1). An aluminum membrane separated the upper from the lower part of the sample and reference cells. A piece of spaghetti 1 cm long (60–100 mg) was set within the winged edge of a vertical stirrer that can be held in the upper part of the cell. Below the aluminum membrane, the cell was filled with 5 ml of water for a pasta-water mass ratio somewhat larger than the minimum required for gelatinization and traditional cooking. The same situation, except for the presence of starchy material, was repeated for the reference cell. When thermal equilibrium was achieved throughout the system, the two stirrers were simultaneously pushed down to pierce the membrane; at the same instant, the calorimetric signal was recorded. Typically, this type of investigation requires a preset output voltage of 0.1 mV and lasts about 1 hr; the record is saved by means of an on-line processor, which allows further data treatment.

Calorimetric scans were carried out at various temperatures in the 70–100°C range; however, the highest operating temperature at which reproducible results can be obtained is 95°C, since when the boiling point is approached, vapor convection causes turbulence within the cells and affects the normal thermal balance between them. However, this temperature limit does not affect the reliability of the results obtained, since gelatinization is very rapid just above 70°C.

Cooking tests. The traditional cooking procedure (D'Egidio et al 1982) was used to prepare samples for comparison with those that underwent the calorimetric scan. Adequate cooking time of these samples was determined by the Braibanti technique, which is based on the disappearance of the uncooked core. This method, which is widely used in Italy, defines the optimal cooking time of pasta as the time required to observe the disappearance of the uncooked core in a small pasta sample manually squeezed

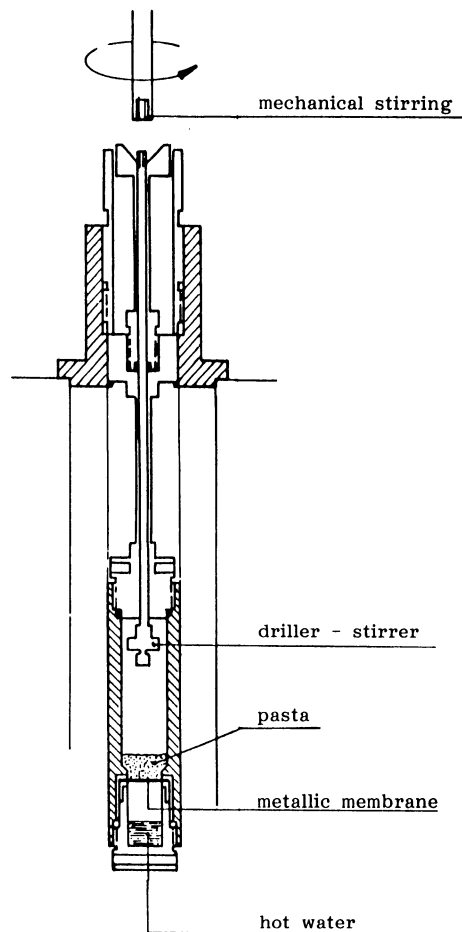


Fig. 1. Mixing vessel with membrane used to simulate the real pasta cooking process; it reproduces the conditions used in investigations on ground pasta.

between two thin Plexiglass plates. The corresponding gelatinization kinetics was defined by measuring at various times the section area of the uncooked core, A_u , and the total section area, A_T , at the polarized light in the microscope. The ungelatinized fraction was assumed to be given by the following:

$$A_{u(t)}\rho_u/[A_{u(t)}\rho_u - A_{g(t)}\rho_g],$$

where $A_{g(t)} = A_T - A_u$, and ρ_u (g/ml) and ρ_g (g/ml) are the densities of ungelatinized and gelatinized region, respectively.

Colorimetric determinations. For a comparison with the results obtained from calorimetric data, the degree of gelatinization was determined according to the Wotton method (Wotton and Chaudhry 1980), that is, via the colorimetric evaluation of the starch-iodine complex formed from either partially cooked or uncooked samples. For this method the gelatinization degree was defined as follows:

$$\alpha = E_t/E_{00},$$

where E_t and E_{00} are the absorbances measured at the time (t) and at the process exhaustion (40 min), respectively.

RESULTS AND DISCUSSION

Treatment of calorimetric data. The output of a differential flux calorimeter is a trace where the signal of the heat power ($S = dQ/dt$) is recorded vs. time (t); the areas underlying peaks thus correspond to heat amounts delivered (exothermic effects) or trapped (endothermic effects) by the system examined.

Isothermal starch gelatinization is accompanied by an endothermic effect that is proportional to the gelatinized mass ($Mg(t)$) at the time (t), as follows:

$$Q(t) = Mg(t) \Delta_{\text{gel}}H = \int_0^t S dt,$$

where $\Delta_{\text{gel}}H$ is the enthalpy of the gelatinization per unit mass. If the total mass of starch that can undergo gelatinization is M , then the gelatinization degree attained at time (t) is: $\alpha(t) = Mg(t)/M$,

and the total heat amount retained will be:

$$Q_{00} = M \Delta_{\text{gel}}H = \int_0^{t_{\text{end}}} S dt,$$

where t_{end} is the time required for the practical exhaustion of the process.

It is easy to recognize that:

$$\alpha(t) = Q(t)/Q_{00},$$

so that the signal S will correspond to:

$$S = Q_{00} d\alpha/dt.$$

If the overall process is assumed to obey a first-order kinetics, as suggested by the trend of our experimental results (see below) and by reliable literature (Lund, 1989), one can write:

$$d\alpha/dt = K(1-\alpha),$$

where K is the kinetic constant. Accordingly,

$$S = K Q_{00} (1-\alpha).$$

This means that the maximum signal, S_{max} , should be observed for $\alpha = 0$, that is, at $t = 0$, thence:

$$S_{\text{max}} = K Q_{00}.$$

One should therefore expect an ideal trace, where an instantaneous signal spike (equal to S_{max}) at $t = 0$ (i.e., when starch and hot water come in contact) is followed by a curve related to the kinetics of the process, as follows:

$$S_{\text{id}} = S_{\text{max}} \exp(-K t).$$

However, the actual trace cannot reproduce such expectations because of the lag time of the thermal diffusion between the sample and the fluxmeters around its cell. In fact, it shows an initial endothermic spike, which is somewhat shifted from the abscissa $t = 0$ and is accorded to a rising curve that gradually reattains the baseline at $S = 0$. When the heat transmission through the cell walls reaches a steady state, the actual signal is expected to parallel the ideal one so as to reproduce the same exponential trend:

$$S = S_0 \exp(-K t),$$

where the preexponential term, S_0 , has no special physical meaning. If heat dissipation is neglected (as with the instrument used in this work), the area underlying the actual trace must be equal to that of the ideal one, as follows:

$$\int_0^{t'_{\text{end}}} S dt = Q_{00},$$

where t'_{end} is the actual time for reattaining baseline. Because of the time lag for thermal diffusion, $t'_{\text{end}} > t_{\text{end}}$; the rising branch of the actual trace is then expected to remain below the ideal curve and its initial spike to be smaller than S_{max} (Fig. 2).

These considerations allow the support of a simple work-line: 1) fitting of the rising branch of the actual trace with an exponential law and evaluation of the kinetic constant, K ; 2) evaluation of

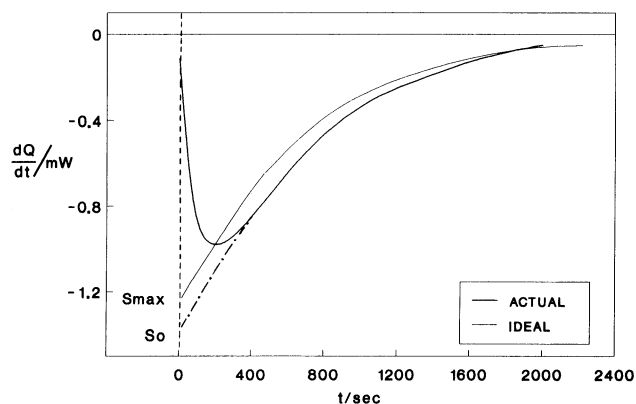


Fig. 2. Calorimetric scan (95°C, durum-HT) of actual and ideal traces. S_{max} = maximal signal, S_0 = preexponential term with no special physical meaning (see text).

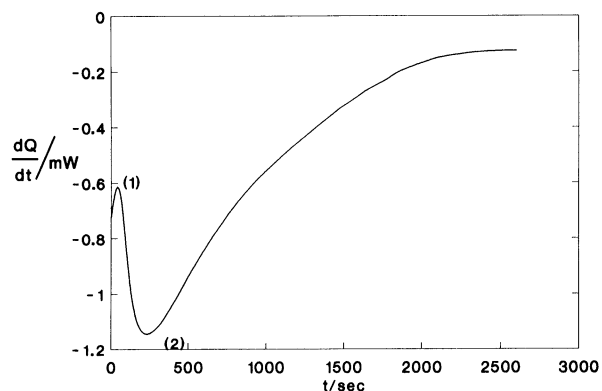


Fig. 3. Typical calorimetric scan (90°C, durum-HT) with exothermic (1) and endothermic (2) peaks. The former is attributed to wetting.

the area underlying the trace so as to attain Q_{oo} ; 3) determination of S_{max} as $S_{max} = K Q_{oo}$; 4) evaluation of t_{end} through the following condition:

$$\int_0^{t_{end}} S_{max} \exp(-K t) dt = Q_{oo};$$

and 5) eventual reproduction of the ideal calorimetric trace that is directly related to the gelatinization process.

The evaluation of the overall thermal effect accompanying the gelatinization process at various temperatures allows a thermodynamic approach based upon the classical Gibbs-Helmholtz relationship:

$$\left[\frac{\partial}{\partial T} \left(\frac{\Delta G}{T} \right) \right]_P = - \frac{\Delta H}{T^2}$$

which can be easily integrated over the range (T_o, T) to yield:

$$\Delta G = \Delta H \frac{T_o - T}{T_o} + T \left(\frac{\Delta G}{T} \right)_{T=T_o}$$

where ΔH was assumed to be independent of temperature.

In the present case, the thermodynamic quantities dealt with the starch gelatinization process, that is, $\Delta G = \Delta_{gel}G$, and $\Delta H = \Delta_{gel}H$, and it is expedient to choose T_o as the minimum temperature at which starch gelatinization is observed, that is, where $\Delta_{gel}G = 0$ can be assumed. Accordingly,

$$\Delta_{gel}G = \Delta_{gel}H \left(\frac{T_o - T}{T_o} \right)$$

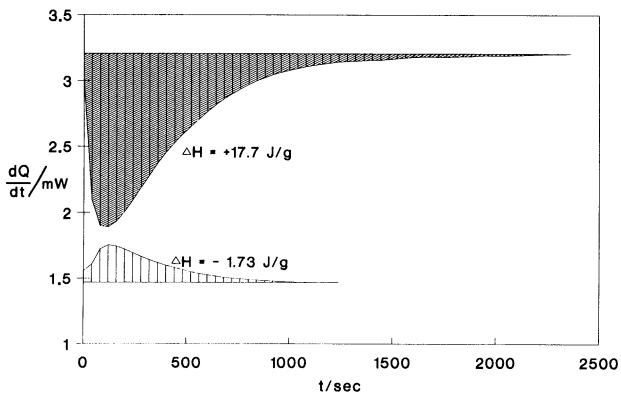


Fig. 4. Calorimetric scan at 95°C (upper curve) and exothermic peak in 30°C run (durum-HT). Enthalpies referred to the weight of the dry sample.

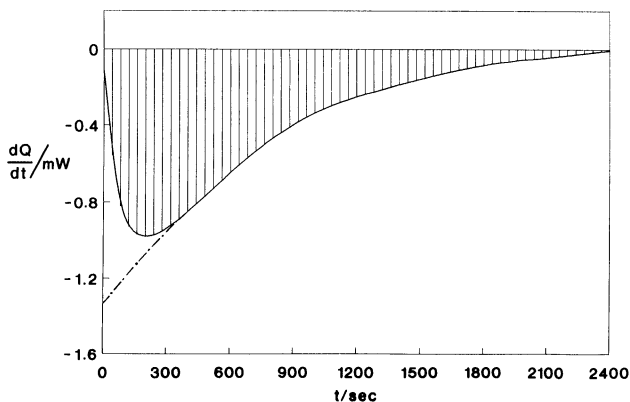


Fig. 5. Isothermal scan with signal integration (95°C, durum-HT), corresponding to a total heat amount of 18 J/g.

Since starch gelatinization is an irreversible process, this expression, which is appropriate only for reversible transformations, cannot be adequate over a wide temperature range. Nonetheless, it satisfies the expectation that $\Delta_{gel}G < 0$ and $\Delta_{gel}G > 0$ for $T > T_o$ and $T < T_o$, respectively, since $\Delta_{gel}H > 0$, and can be considered rigorous in the proximity of $T = T_o$.

The isothermal calorimetric determinations at various temperatures allow us to state that, for systems with a starch-water mass ratio of < 0.1 , T_o should be close to 333 K. The derivative of this $\Delta_{gel}G$ function allows evaluation of the entropy change for starch gelatinization, as follows:

$$\Delta_{gel}S = - \frac{\partial}{\partial T} (\Delta_{gel}G) = \Delta_{gel}H / T_o \approx 0.05 \text{ J/g K}$$

where $\Delta_{gel}H$ was again assumed independent of temperature, and starch gelatinization was treated as a reversible process in the proximity of $T = T_o$.

A possible complication in the treatment of these isothermal calorimetric traces comes from an early exothermic effect, which sometimes precedes the endothermic spike of the gelatinization process. The exothermic peak can be attributed to the wetting of the starch, that is, the water sorption at the surface of the starchy material (Shiotsubo and Takahashi 1986). It can be satisfactorily singled out by pouring the sample into water kept at $T < T_o$, where no gelatinization occurs. This allows us to assess whether the heat involved is negligible with respect to the following endothermic gelatinization, and/or whether or not the duration of the process affects the rising branch of the gelatinization signal, which is crucial for the data treatment. In the present work, starch wetting was quantitatively characterized, and any significant influence on the gelatinization peak was ruled out.

Experimental results. Figure 3 shows a typical trace obtained in the present work, where both the early exothermic and the main endothermic peaks are present. Figure 4 shows the quantitative resolution of the two effects, where the exothermic peak was singled out from an isothermal scan at 30°C. Since its enthalpy accounts only for 10% of the endothermic signal and has a poor influence on the rising branch of the latter, it was disregarded

TABLE I
Kinetic Constant (K) in Five Runs of Calorimetric Experiments with Durum-HT Spaghetti in Original Shape

Run at 95°C	$K \times 10^3/\text{sec}$
1	1.618
2	1.639
3	1.603
4	1.615
5	1.631
Average	1.621
SD	0.014

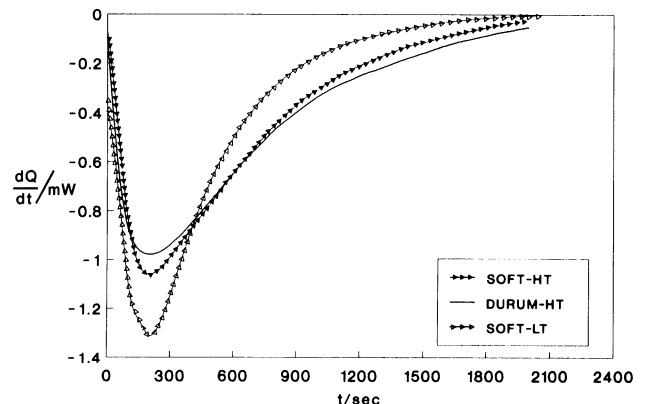


Fig. 6. Calorimetric scan (95°C) of different types of pasta.

TABLE II
Relationship Between Temperature and Kinetic Constant (*K*) in Experiments with Durum-HT Spaghetti in Original Shape

Temperature (°C)	<i>K</i> × 10 ³ /sec
70	0.434
80	0.856
85	1.137
90	1.440
95	1.631

TABLE III
Kinetic Constant (*K*) in Four Runs of Chemical Determinations of Gelatinization Degree (Durum-HT Spaghetti, 100°C)

Run	<i>K</i> × 10 ³ /sec	<i>R</i> ²
1	2.79	0.962
2	2.12	0.986
3	2.69	0.994
4	2.04	0.992
Average	2.41	...
SD	0.38	...

throughout the present data treatment.

The fit of the rising branch of the endothermic signal with an exponential law did not allow us to observe any significant (*t'*_{end} - *t*_{end}) difference because of the very flat trend of both traces at the end of the process. This justified the assumption that the "real" rising branch of the endothermic signal can be considered very close to the ideal trend.

Figure 5 shows the total area, *Q*₀₀, swept by the endothermic trace in a typical scan. The reproducibility of the calorimetric method can be verified by inspection of the values of the kinetic constant reported in Table I; these are relevant to five runs carried out on different samples of commercial durum-HT raw spaghetti examined at 95°C in the original shape. The small standard deviation and the high correlation coefficient (*R*² > 0.99) in any run support the reliability of the results. These data allow us to argue that the cooking time recommended by the manufacturer (11 min) corresponds to a gelatinization degree of 0.65, while 0.99 gelatinization is attained after about 50 min of cooking.

Figure 6 shows the behaviors of the various kinds of pasta examined in the present work. The corresponding *K* values (× 10⁻³s⁻¹) at 95°C are 1.631, 2.000, and 2.970 for durum-HT, soft-HT, and soft-LT, respectively. These values agree with the traditional experience of longer cooking times for durum-HT and can be justified by the classic observation of different network structurization and by the influence of the drying conditions used in the industrial process (Resmini et al 1988, Piazza et al 1990b). Calorimetric scans performed at various temperatures were treated in the same way and correspond to the kinetic constant reported in Table II. If these data are worked out according to the Arrhenius relationship, *K* = *K*₀ exp (-*E*_a/RT), an "activation energy" can be calculated:

$$E_a = 61.4 \text{ kJ/mol},$$

which can be compared with some data in the literature (Lund 1989). It is obvious that, due to the narrow temperature range examined, the reliability of these *E*_a values, as well as of the Arrhenius relationship itself, is largely doubtful. Nonetheless, it can be used to compare the results of different authors as well as ours to evaluate *K* at 100°C, that is, 2.5 × 10⁻³/sec. However, in these cases one should also take into account that sets of experimental data in good agreement with one another could correspond to values of the preexponential, *K*₀, and activation energy terms that differ among authors. This depends on the mutual compensation between *K*₀ and *E*_a, so that large *E*_a and *K*₀ can yield a fit as good as that of a small *E*_a with a small *K*₀.

The value of the kinetic constant obtained from ground durum-HT spaghetti treated at 95°C was 1.97 × 10⁻³/sec. This result

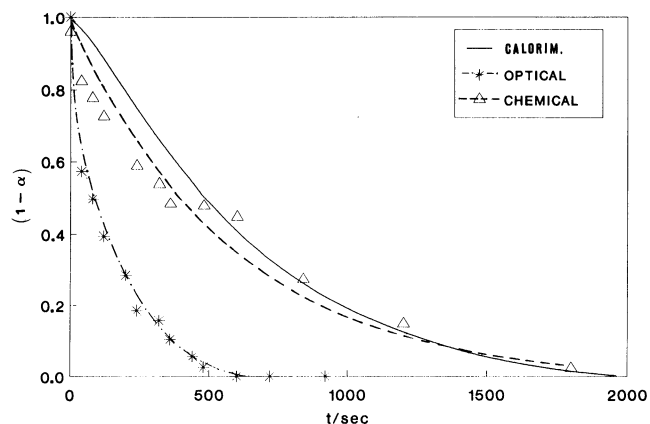


Fig. 7. Comparison of gelatinization kinetics measured by different methods (durum-HT, 100°C).

exceeds by about 20% the corresponding values obtained from spaghetti cooked in the original shape (Table I). The calculated difference may be attributed to the quicker water diffusion into the ground samples and, as a consequence, to the smaller time required to wet the starch granules.

K values drawn from the classic chemical approach (see materials and methods section) and referred to durum-HT spaghetti cooked at 100°C (Table III) correspond to four different runs. In this case, the data are rather scattered, as confirmed by the low correlation coefficients. Furthermore, the calculated standard deviation does not allow satisfactory confidence in the results. Analogous conclusions came from the results of the optical method, which gave an even poorer *R*² value (0.92).

CONCLUSIONS

Assessment of the gelatinization degree as well as the relevant kinetic parameters is of utmost importance to characterize the overall quality profile of pasta products. Isothermal investigations carried out with a differential flux calorimeter allowed quantitative and reliable assessment of the kinetic parameters of starch gelatinization in the pasta cooking process. The operating conditions set for the present study (which were rather similar to those of the actual cooking process) and the satisfactory reproducibility of the experimental data justify good confidence in the results obtained. Indeed, these results showed a much more regular trend of gelatinization degree vs. time than the results obtained from other approaches (Fig. 7). This is partly due to the fact that calorimetry allows continuous isothermal scans within the time range considered; nonetheless, the single-point measurements relevant to the other approaches would not provide fits of comparable reliability because the experimental uncertainty was much greater. Further investigations are in progress and aim to compare different shapes of pasta products as well as different process technologies. Furthermore, work has been undertaken to compare hydration and gelatinization kinetics.

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