

Starch Retrogradation in Cooked Pasta and Rice

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ABSTRACT

Cereal Chem. 77(4):433–438

Effect of cooking time on starch retrogradation and water distribution was studied in pasta (spaghetti) and rice (parboiled and arborio) using differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA). Optimum cooking times (OCT) were 8, 16, and 18.5 min for spaghetti, parboiled, and arborio rice, respectively. Swelling was observed by image analysis. OCT spaghetti and rice showed various starch retrogradation rates at various aging times and temperatures. Based on the classical Avrami function, the retrogradation rate at 5°C followed the order spaghetti > parboiled rice > arborio rice, while extent was in the opposite order. At higher temperature (20°C), the rates decreased by 20×

in all cases. Thermogravimetric analysis (TGA) investigations were undertaken to check the distribution of water within these products and its relationship to starch retrogradation. During heating, water was released in two distinguishable steps at ≈80 and 100°C. Results supported the conclusion that the more tightly bound water might not participate or facilitate starch retrogradation. In this study, the overall water content did not change during storage, and water appeared to migrate from sites of stronger binding to sites of weaker binding. The temperature dependence of the Avrami constant was described with the Vogel-Tamman-Fulcher empirical expression.

Pasta and rice are very popular cereal products with an important role in the Mediterranean diet, either as warm dishes or as basic ingredients of salads. These products are also used in precooked meals with a rather short shelf-life. In many cases, boiled pasta and rice are stored at subambient temperatures to be used some hours later in the preparation of the meal.

Following cooking, the starch component of these products undergoes retrogradation, defined as partial crystallization of amylopectin within the gelatinized starch fraction, with an increase of firmness and a modification of taste (Piazza et al 1994, Fan and Marks 1998, Ohya and Kawabata 1998). A mild heat treatment allows the sensory properties of the freshly prepared product to be fully recovered, but this requires careful control to avoid overcooking, which tends to further gelatinize the starch, resulting in a product that is too soft and sticky. In the case of pasta- or rice-based salads, no reheating is done, and aging severely reduces the sensory qualities of the product, mainly because of starch retrogradation.

Starch retrogradation has been well described and studied in starch gel and in bread crumb samples (Eliasson 1985, Zobel 1988, He and Hosney 1990, Piazza and Masi 1995, Schiraldi et al 1996, Fessas and Schiraldi 1998) because it is an important component of bread staling. However, much less has been published about starch retrogradation in boiled pasta and rice (Resmini and Pagani 1983, Fan and Marks 1998). Some authors recognized the role of water and its mobility (He and Hosney 1990, Piazza and Masi 1995). In the case of bread crumb, a mechanism has been proposed (Schiraldi et al 1996) to describe the behavior of water that may facilitate or influence inter-chain bridging. The present work involved a study of the extent and the rate of starch retrogradation in pasta and rice as affected by storage temperature. Calorimetry, thermogravimetry, and image analysis investigations were used to define an optimum cooking time (OCT) and progress of starch retrogradation in OCT products stored at various temperatures for different time periods. The quantitative role of water on starch retrogradation was also investigated.

MATERIALS AND METHODS

The products considered were commercial samples of durum wheat spaghetti, parboiled rice, and arborio rice. Raw products were analyzed according to Approved Methods (AACC 2000) and with image analysis.

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Cooking Tests

Raw pasta or rice (100 g) was mixed with 1 L of tap water and cooked without addition of salt (D'Egidio et al 1982). The cooking process was stopped at various times by quenching the product in ice water for 5 min, draining, and resting on filter paper for 3 min. The water uptake of the cooked product was determined in three replicates by weighing before and after the cooking process. The calculated mass ratio between cooked and uncooked samples (W/W_0) varied within ± 10% error. Some cooked samples were freeze-dried (Lioflex 04, Edwards, UK) before evaluating the residual ungelatinized starch fraction, which was done after rehydrating the freeze-dried sample to a known moisture content.

Image Analysis

Swelling of product during the cooking was monitored by examining digital cross-sectioned images of cooked and drained spaghetti and rice kernels (Spagnolello and Riva 1996) obtained by directly scanning the product (Deskscan Jet IIc, Hewlett Packard). Sectioned samples were placed on the glass plate of the scanner with a non-reflecting black paper sheet covering to facilitate the automatic detection of the contours. Typically, 100 rice kernels and 20 cross-sectioned spaghetti samples were analyzed for each cooking time.

A 1,200 pixel/in. resolution (for spaghetti cross section) and 300 pixel/in. (for rice kernels) was chosen. Images (recorded in a gray level scale) were acquired and automatically analyzed (Image Pro Plus 3, Media Cybernetics) using a built-in threshold procedure (automatic bright objects) before making a determination of shape, maximum and minimum diameter, and area. Area was quantified and expressed as A_i/A_0 ratio (A_0 being the cross section area of uncooked sample). This was presented because it is considered proportional to the overall swelling for both products (Spagnolello and Riva 1996).

DSC Investigations

Differential scanning calorimetry (DSC) was conducted using a Perkin-Elmer DSC 6. The temperature range was -15 to 110°C, and dry N₂ was used as the purging gas. Calibration was performed with indium at the experimental heating rates of 2 and 5°C/min for retrogradation and gelatinization studies, respectively. Sealed 140-μL aluminum DSC pans were used for all the investigations. The typical sample mass was 25 mg of freeze-dried product (residual moisture <2%) added to 25 μL of distilled water for gelatinization and 35 mg of cooked product for retrogradation.

For starch gelatinization, the prepared samples had a well-defined excess water content above the threshold for a complete gelatinization (Zeleznek and Hosney 1986, 1987). No substantial effect on the starch melting endotherm due to evaporation was using the sealed DSC pans.

RESULTS AND DISCUSSION

Retrogradation tests were performed with OCT samples stored under vacuum in sealed plastic bags (layered polyamide and polyethylene sheet, Tillmans, Milano, Italy). Each bag contained 20 g of cooked product. The products were stored in thermostatic cells set at -5°C (spaghetti only), 5, 10, and 20°C with a $\pm 1^{\circ}\text{C}$ variation up to 20 days. Aluminum foil pieces with a comparable heat capacity were sealed in the reference pan. Each analysis was done at least in duplicate.

The heat flow signals were recorded in an ASCII format and analyzed in a personal computer with Table Curve and Peakfit software (Jandel). The data analyses (base-line assessment, trace smoothing, and trace deconvolution) were performed as reported previously (Riva and Schiraldi 1993). The gelatinization and retrogradation (fusion of amylopectin crystals) signals were typically represented by endothermic peaks. Peak area was used to evaluate the enthalpy (Schiraldi et al 1996).

TGA Investigations

Thermogravimetric (TGA) determination of water losses was made using a TG-DSC 111 (Setaram, Lyon, France) operating with open pans. Two open ampoules suspended from the arms of a balance were hung within the parallel cylindrical cavities of a twin Calvet calorimeter (Schiraldi et al 1996). During heating (at a given heating rate), heat flux and balance shift are simultaneously recorded. However, because the vaporization of water is predominate over all other thermal effects, the data mostly represented the water loss on heating. Samples (40 g) were tested at $2^{\circ}\text{C}/\text{min}$ over a $20\text{--}120^{\circ}\text{C}$ temperature range with an empty cell as a reference.

Data were analyzed using the same software as for DSC investigations. The experimental data were fitted with two sigmoid functions to attain a closer description of the actual trace.

When the time derivative (DTG) of the TGA record was considered, a double peak was obtained that was fitted using the two derivative functions (for each sigmoid function). The corresponding integral reflected the total amount of water released (Schiraldi et al 1996).

TABLE I
Relevant Characteristics of Pasta and Rice Commercial Samples

Rice	Spaghetti	Parboiled Rice	Arborio
Brand type	Barilla, Spaghetti n. 5 diameter	Flora, Fino Ribe "il classico" kernel volume	San Marco, Superfino kernel volume
Size	$1.73 \pm 0.033 \text{ mm}$	$26.4 \pm 3.2 \text{ mm}^3$	$37.4 \pm 5.6 \text{ mm}^3$
Moisture (%)	10.0 ± 0.2^a	11.9 ± 0.3	12.8 ± 0.3
Ash (g/100 g) ^b	0.99 ± 0.04	0.70 ± 0.04	0.60 ± 0.03
Protein (g/100 g) ^b	14.78 ± 0.25	7.20 ± 0.10	7.00 ± 0.15
SCT (min) ^c	8	18	18–20

^a Average data \pm standard deviation drawn from five replicates.

^b Moist mass of uncooked product.

^c Suggested cooking time from packaging label.

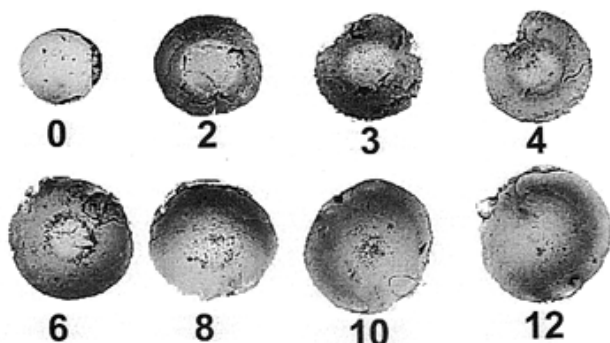


Fig. 1. Selected images of spaghetti cross sections at various cooking times (in minutes)

Retrogradation can occur within pregelatinized starch like that present in bread and boiled potatoes, pasta, and rice (Eliasson 1985, He and Hosoney 1990, Piazza and Masi 1995, Schiraldi et al 1996, Fan and Marks 1998, Fessas and Schiraldi 1998). The extent of retrogradation largely depends on the molecular and structural nature of gelatinized starch, which is highly influenced by the moisture, thermal history, and water content of the product. Its rate is rather low with a maximum at $\approx 5^{\circ}\text{C}$ and 50% water content in starch gels (Zeleznaek and Hosoney 1986, 1987). For this reason, pasta and rice samples were previously boiled in excess water for a well-defined cooking time to carefully assess the gelatinized starch fraction.

OCT

Table I summarizes the relevant characteristics of uncooked products. Boiled pasta and rice are swollen and heavier than the uncooked products because they adsorbed large amounts of water. This was observable by eye and by image analysis (Fig. 1). Although more accurate methods (D'Egidio and Nardi 1996) are available, the mass increase and the size modifications were sufficient to assess the cooking progress of these starch products. Figure 1 shows the comparison between images obtained at various cooking times for spaghetti. Figure 2 shows the changes in DSC gelatinization endotherms that decreased in magnitude with increased cooking time. The area underlying the signal was used to calculate the amount of starch fraction that remained ungelatinized (Riva et al 1994). The results are as expected for gelatinization of starch products in excess water. Onset and conclusion temperatures occurred at ≈ 50 and 80°C , respectively. Figures 3–5 show W/W_0 , A/A_0 , and $\Delta H/\Delta H_0$ values for spaghetti, parboiled rice, and arborio rice, respectively. In Fig. 3, A/A_0 , the area increase of spaghetti cross sections and rice kernels (related to the volume increase accompanying the water uptake), showed two types of behavior with an intermediate knee that can be explained as a partial collapse of the product. This was more observable in spaghetti than in either of the rice samples. The OCT was defined as the cooking time at the knee point. Before OCT was reached, gelatinization and swelling are responsible for the increased A/A_0 ; after OCT, A/A_0 could be characterized as water accumulation sustained by a diffusion process through the gelatinized system (Piazza et al 1994). The intersection of these trends corresponds to the flex point of the overall mass increase, W/W_0 . In these conditions, the product behaved like a sponge where water uptake produces a mass increase with negligible volume

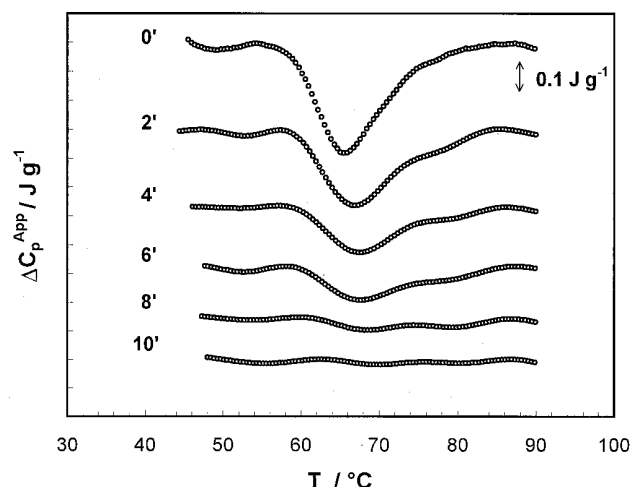


Fig. 2. Differential scanning calorimetry (DSC) results of partially cooked spaghetti (cooking time in minutes). Signal is reported as apparent heat capacity and referred to unit mass of dry matter.

change. At this time, the ungelatinized starch fraction (α_{ungel}) was small, although not identical, among the three products. According to the first order kinetics:

$$\alpha_{\text{ungel}} = 1 - \exp(-k \times \text{OCT}) \quad (1)$$

where the estimated values of the kinetic constant (k) were 4.4, 1.3, and $2.7 \times 10^{-3} \text{ sec}^{-1}$ for pasta, parboiled rice, and arborio rice, respectively.

The α_{ungel} was greater for parboiled than for arborio rice at OCT of 16 and 18.5 min, respectively. This was as expected in a parboiling process when starch-protein interactions and tightening of the kernel structure might have occurred (Nardi et al 1994). These OCT seemed to be in agreement with the suggested cooking time (SCT), and OCT samples were used as references for samples in retrogradation studies.

Starch Retrogradation

OCT boiled pasta and rice were sampled after various aging times at various temperatures ($-5, 5, 10,$ and 20°C) for DSC investigations to determine the fusion enthalpy of the amylopectin crystals formed during storage. Figure 6 shows the results obtained for cooked parboiled rice kept at 10°C . Similar results were found for spaghetti and arborio rice at all storage temperatures. The trend of the endothermic effects was described with an Avrami fitting function:

$$\Delta H_t = \Delta H_\infty [1 - \exp(-k \times t^n)] \quad (2)$$

where ΔH stands for fusion enthalpy (at storage time t and ∞); the exponent $n = 2$ (according to regression analyses) for all the samples examined; this choice came from the observation that the Avrami constant k and the exponent n appeared correlated. Figure 7 shows the ΔH increasing with increasing time and decreasing temperature for parboiled rice. Table II summarizes the results of the Avrami parameterization for all three products. As expected, pasta and rice samples stored at 5°C underwent a more extended starch retrogradation; however, significant differences were observed between pasta and rice. At 5°C , the retrogradation rate order was spaghetti > parboiled rice > arborio rice, but the degree of retrogradation was in the opposite order,

arborio rice > parboiled rice > spaghetti. This could be explained by the differences in protein content (spaghetti had a large gluten fraction), amylose-amylopectin mass ratio (higher amounts of amylose in rice), and previous thermal treatment (spaghetti is heat-extruded and dried; parboiled rice is partially gelatinized and restructured by steam). These could be related to the available moisture that has been known to directly affect the mechanism of starch retrogradation at the molecular and microscopic levels.

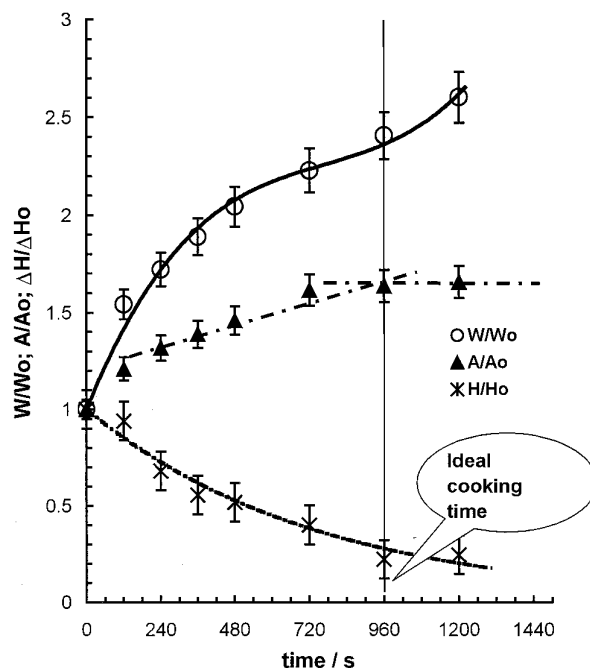


Fig. 4. Cooking kinetics of parboiled rice: trends of mass increase (W/W_0), swelling (A/A_0), and ungelatinized starch fraction (gelatinization enthalpy, $\Delta H/\Delta H_0$).

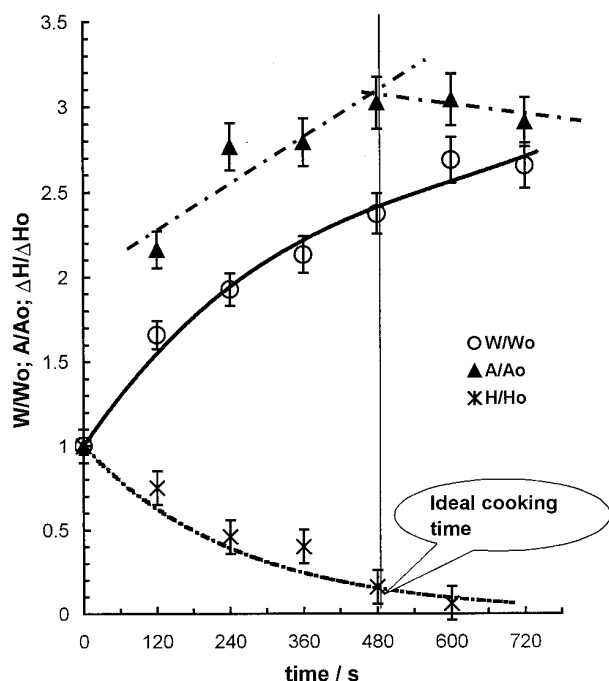


Fig. 3. Cooking kinetics of spaghetti: trends of mass increase (W/W_0), swelling (A/A_0), and ungelatinized starch fraction (gelatinization enthalpy, $\Delta H/\Delta H_0$).

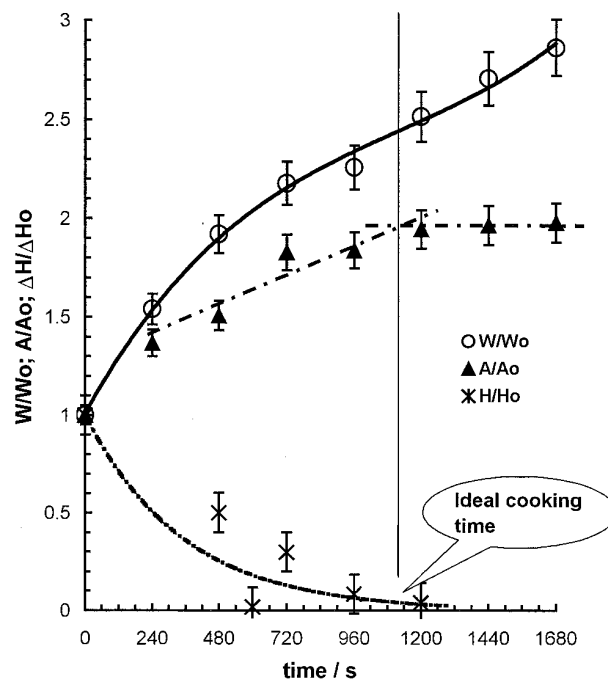


Fig. 5. Cooking kinetics of arborio rice: trends of mass increase (W/W_0), swelling (A/A_0), and ungelatinized starch fraction (gelatinization enthalpy, $\Delta H/\Delta H_0$).

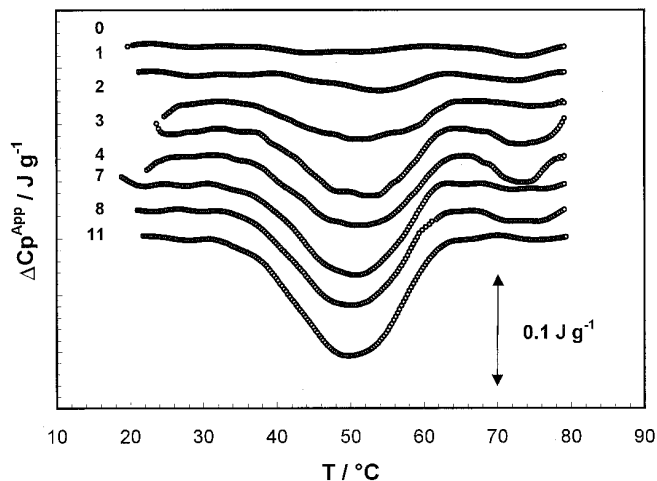


Fig. 6. Differential scanning calorimetry (DSC) endotherm of amylopectin fusion in retrograded parboiled rice. Storage temperature 10°C; storage time (in minutes) is given beside each trace.

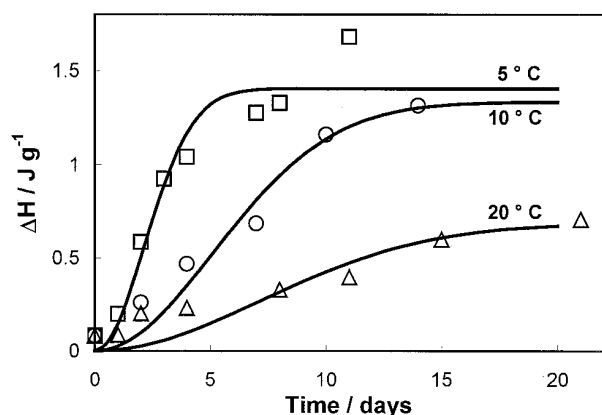


Fig. 7. Avrami fitting of starch retrogradation in cooked parboiled rice stored at various temperatures.

Available Moisture

TGA investigations allowed determination of the water uptake per gram of dry matter in cooked products, which is practically the same for pasta (65.2%) as for rice (64.6%). In addition, this technique was applied to investigate the availability of water within cooked and aged products as it plasticizes and influences the chain-to-chain alignment underlying formation of the crystalline structure and nuclei (Bushuk and Hlynka 1964, Slade and Levine 1993, Schiraldi et al 1996). TGA data can be adequately used when the DTG of the relevant record is considered (Schiraldi et al 1996). The DTG trend clearly showed a broad peak that was deconvoluted into a couple of simple (Gaussian) components with well-separated maximum temperatures at ≈ 80 and $100 \pm 5^\circ\text{C}$, respectively. Figure 8 shows the results obtained for spaghetti.

Water could be considered therefore to be distributed in at least two distinguishable populations (w_1 and w_2) for the low and high temperature DTG maximum, respectively. The water amounts were obtained from the integrals of the corresponding DTG peaks. The mass ratio (w_1/w_2) presented in Fig. 9 went through a broad maximum occurring at an aging time dependent on storage temperature for spaghetti and parboiled rice stored at 5 and 20°C. These results also supported the trend observed from the Avrami-fitted functions for amylopectin fusion enthalpy. Figure 9 shows that, for both products, the w_1/w_2 maximum corresponded to the retrogradation rate that also went through a maximum.

A similar correlation was observed for bread staling (Schiraldi et al 1996) where the w_1/w_2 went through a minimum. At 5°C, it

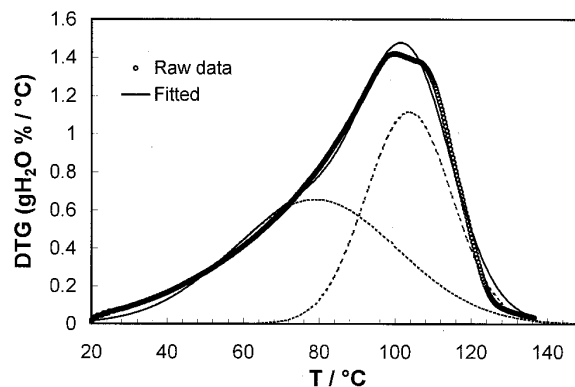


Fig. 8. Time derivative (DTG) of thermogravimetric analysis (TGA) record obtained at 2°C/min scanning rate on spaghetti stored eight days. Dotted and dashed lines represent deconvoluted components of the DTG record.

can be observed that the w_1/w_2 maximum occurs earlier in spaghetti than in parboiled rice, although in the latter case it attained a larger value. The presence of the w_1/w_2 maximum suggests that water can migrate from sites where it is bound more strongly to sites where it is more weakly bound. Perhaps water is more tightly bound to proteins than to the starch gel. This means that w_1 may be related to the water retained by the starch gel, therefore, directly affecting the starch retrogradation. The w_2 may be related to the moisture associated with the proteins. The larger w_1/w_2 maximum attained in rice samples could be explained by the smaller protein content as compared with spaghetti (Table I).

The onset of starch retrogradation depends on a number of factors, including water mobility within the starch gel and amylose-amylopectin mass ratio. Because the w_1/w_2 maximum in spaghetti samples was reached earlier than in rice, it might be suggested that water was more mobile in spaghetti than in rice, based on the difference in amylose-amylopectin ratio.

Mechanical tests (data not shown) aimed at determining the energy to compress and extrude spaghetti and rice samples were in line with those from DSC and TGA investigations. The mechanical data did not support any interpretation of starch retrogradation but that the load required to attain a given deformation increased with the storage time and therefore with the extent of starch retrogradation. However, this does not exclude other factors that may be involved. As suggested earlier (Pagani et al 1986, Eliasson and Larsson 1993), starch-protein interactions might also be responsible, but no specific experimental evidence has been reported.

Effect of Temperature

Figure 10 shows the fit of the Avrami constant versus temperature for the spaghetti samples. It was clear that the classical Arrhenius expression could not fit the data satisfactorily. This was expected because the formation of amylopectin crystals shows a bell-shaped trend (Zeleznaek and Hosney 1986) with fusion temperature (T_m) = 0. The maximum rate occurs at an intermediate temperature between T_m ($\approx 50^\circ\text{C}$) and T_g that depends of the water content of the sample. In this temperature range, the rate of nucleation and growth of crystal phases depend on the molecular mobility and show a bell-shaped trend versus temperature. This is the result of the composed probability of two independent events: formation of nuclei and growth of crystals (Slade and Levine 1991), the rates are temperature-dependent. Because the systems considered here are polymers, it seemed reasonable to describe the relevant $k(T)$ trend using the empirical Vogel-Tamman-Fulcher law (Angell and Sichina 1976) in a suitably modified form:

$$k = k_0 \exp\left(\frac{B}{T - T_0}\right) \quad (3)$$

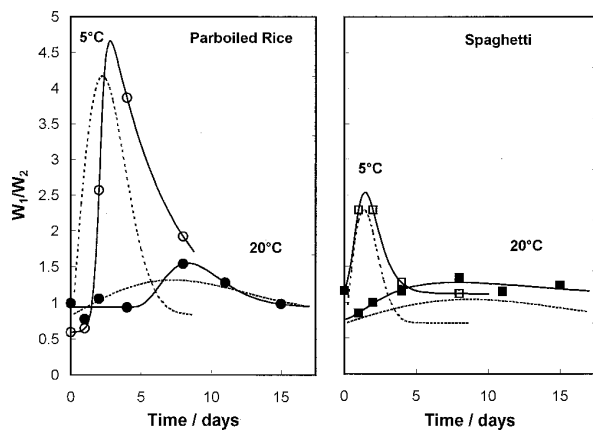


Fig. 9. Trend of water mass ratio w_1/w_2 during of parboiled rice (left) and spaghetti (right) compared with the trend of the time derivative (in arbitrary units) of the Avrami equation that fits the relevant amylopectin fusion enthalpy (dotted line). Storage at 5°C (open symbols) and 20°C (filled symbols)

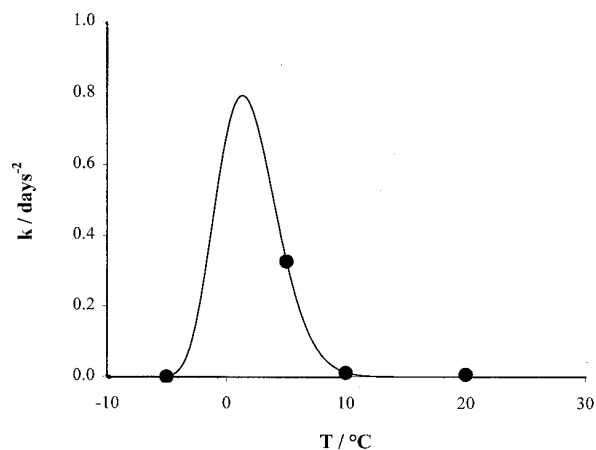


Fig. 10. Avrami constants (filled symbols) as a function of storage temperature for cooked spaghetti. Fitting curve (line) corresponds to Eq. 4.

This equation is valid in a wide temperature and viscosity range not too close to the glass transition temperature (T_g) and could be applied to the constants of either processes:

$$k_{overall} = k_{nucl} \cdot k_{growth} = k_{0,nucl} \cdot \exp\left(\frac{B_{nucl}}{T - T_{0,nucl}}\right) \cdot k_{0,growth} \exp\left(\frac{B_{growth}}{T_{0,growth} - T}\right) \quad (4)$$

In either terms, T_0 is a suitable reference temperature at which the corresponding constant vanishes. Within the temperature range of interest, $T_{0,nucl}$ and $T_{0,growth}$ are the lower and the upper limit, respectively. They could be tentatively identified with the T_g of the still amorphous system and the melting point of the crystals, respectively. The parameters B_{nucl} and B_{growth} might be related to some activation energy (in R units, the gas constant) of either process.

Because a reliable value of T_m had been experimentally determined as $T_m = 50 \pm 3^\circ\text{C}$, this value was used to replace $T_{0,growth}$ in Eq. 3. In spite of the number of independent parameters in the fitting equation, a satisfactory fit of the experimental $k_{overall}$ data (Fig. 10) could be achieved only when $T_{0,nucl}$ was given a value of $-10 \pm 3^\circ\text{C}$. This value was also in the expected T_g range for a starch gel containing a similar moisture content.

The maximum of the bell-shape trend occurred at 2°C , which was close to the temperature of maximum retrogradation rate reported for starch gels (Zeleznek and Hosney 1986).

TABLE II
Relevant Avrami Parameters of Pasta and Rice Commercial Samples

	Spaghetti	Parboiled Rice	Arborio Rice
-5°C			
k (days ⁻²)	$1.3 \cdot 10^{-4}$		
5°C			
k (days ⁻²)	$3.27 \cdot 10^{-1}$	$1.11 \cdot 10^{-1}$	$2.79 \cdot 10^{-2}$
ΔH_∞ (J/g)	0.225	1.404	1.640
10°C			
k (days ⁻²)	$1.16 \cdot 10^{-2}$	$1.91 \cdot 10^{-2}$	
ΔH_∞ (J/g)	0.919	1.333	
20°C			
k (days ⁻²)	$6.73 \cdot 10^{-3}$	$9.69 \cdot 10^{-3}$	$<1 \cdot 10^{-3}$
ΔH_∞ (J/g)	0.324	0.686	<0.3

CONCLUSIONS

Starch retrogradation in cooked spaghetti and rice was studied by combining two thermal analyses from DSC and TGA. It was observed that retrogradation was faster in spaghetti but less extensive. The DTG suggested that water might be present in at least two populations with different mobility. Accordingly, higher mobility but less water would be available to sustain starch retrogradation in spaghetti than in rice samples, thus leading to a more rapid rate and smaller extent of retrogradation.

According to the results of the present work, starch retrogradation in boiled pasta and rice would be lower when the product is stored at room temperature, avoiding moisture exchanges in sealed bags, than in a refrigerator (as often observed in catering practice). The actual choice of the storage temperature should, however, also account for prevention of microbial spoilage. It should be also emphasized that the starch retrogradation can be beneficial in cases where the nutritional benefit of resistant starch is desirable (Casiraghi et al 1992, Björck 1996).

Products with larger protein content might interact with water more tightly, reducing its more mobile fraction that seems directly related to starch retrogradation. The role of other wheat components like arabinoxylans and arabinogalactans (pentosans), that can fix water (Fessas and Schiraldi 1998) remain to be investigated. As a consequence, the quality of wheat used to prepare pasta could affect its tendency to retrograde.

Thermal treatments like extrusion, drying, and parboiling can affect the structure of the product, and therefore the mobility of water uptake in the process, as well as the rate of starch retrogradation.

ACKNOWLEDGMENTS

Research supported by FAIR CT 96 - 1085 European Project.

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[Received June 2, 1999. Accepted March 20, 2000.]