

Effect of Sugars on Retrogradation of Waxy Maize Starch-Sugar Extrudates

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ABSTRACT

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The effect of sucrose, fructose, and xylose on the retrogradation of waxy maize starch extrudates at relatively low moisture contents (20–50 g of water/100 g of dry solid) at 277–353 K was investigated using X-ray diffraction and ¹H nuclear magnetic resonance (NMR) relaxometry. The role of the sugar depended on the type of sugar and its concentration, but most importantly on the retrogradation conditions (water content and storage temperature). For the isothermal retrogradation at 313 K, fructose considerably increased the rate of retrogradation over the range of water contents investigated, and the increase was proportional to the sugar concentration. The behavior of xylose and sucrose was more complex. At 10%

sugar content, both sugars enhanced starch retrogradation, while at 30% sugar content, xylose accelerated the process at <35% (mc, dsb) but decreased the rate constant when more water was present. This crossover took place at 42% mc for sucrose. Sugars enhanced the transformation of the A-type polymorph to a so-called pseudo-B with an X-ray diffractogram similar to that of B-type starch, with the exception of the 1.6-nm peak which was considerably depressed. The effects of these sugars on the retrogradation kinetics in variable storage temperature conditions mirrored those obtained when isothermal retrogradation was examined in the water content domain.

The impact of sugars and other small and intermediate-sized molecules on the properties of polymers is of great interest for the polymer, pharmaceutical, and food industries. Particular attention was given to the ability of these small and intermediate molecules, particularly water and sugars, to plasticize macromolecules such as proteins and starch polysaccharides (Kalichevsky et al 1993, Roos 1995). The impact of additives on the recrystallization of gelatinized amorphous starch is of considerable importance for the understanding of the processing and the shelf-life of many starch-based foods (baked products, breakfast cereals, snacks, etc.) where sugars, lipids, flavors, and salt, etc., are added to improve the perception of the final product and sometimes to control the staling process. However, the control of the retrogradation process is still empirical and often ineffective.

Miura et al (1992) studied the effects of various polyols and emulsifiers on the retrogradation rate of nonglutinous rice starch. Their results suggested that such additives retarded the retrogradation process, and suggested that this effect resulted from the formation of a water-structure in the presence of polyols and of specific interactions between the starch chains and the emulsifier molecules. The effect of added sugars on the retrogradation of starch has been investigated by several workers, but the results were not always consistent (Marsh 1986, Cairns et al 1991). While Marsh (1986) found that sucrose and maltose delayed the retrogradation of wheat starch gels, fructose enhanced it, and xylose inhibited the amylopectin recrystallization process, Cairns et al (1991) reported that glucose slowed the retrogradation while sucrose and ribose inhibited entirely the retrogradation of similar wheat starch gels.

The food polymer science approach pioneered by Slade and Levine (1991) based on the exploitation of the relationship between the glass-rubber transition and the degree of molecular mobility and, subsequently, the rate of chemical and physical changes offered a new opportunity to rationalize the effect of various additives on the kinetics of starch retrogradation. Wang et al (1994) reported the enhancing effect of sugars and maltodextrins on the retrogradation of starch and correlated the results with the glass transition temperature (T_g) of the system. Biliaderis and Prokopowich (1994) studied the effect of polyhydroxy compounds (PHC, including various sugars, glucose oligomers G1–G8, and polyols) on the kinetics of retrogradation of concentrated waxy maize starch gels. They subsequently have extended the study to wheat, potato, and pea starches

(Prokopowich and Biliaderis 1995). Their findings for waxy maize starch, PHC, and water systems (1:0.5:1.5) at a storage temperature of 6°C suggested that: 1) for the glucose oligomers, maltotriose inhibited the recrystallization of amylopectin, while the sample containing maltooctaose showed the highest extent of starch retrogradation; 2) in contrast, glucose and fructose enhanced the retrogradation rate in the sugar series, while ribose and xylose retarded the process. The authors attempted to relate the effect of each sugar on the retrogradation kinetics to some of its physicochemical properties (hydration number, isentropic partial molar compressibility, rotational correlation time, and relative mobility). This approach provided some insight into the dependency of the effect on the type of sugar but offered little insight into the dependency on the sugar concentration. For example, fructose seemed to slightly inhibit the retrogradation at concentrations <7.5% (w/w) but had the opposite effect at higher concentrations, while the inhibiting effect of ribose increased steadily with increased sugar concentration. Finally, a concern that could be raised about a large proportion of the published work on the effect of additives such as sugars on starch gelatinization and subsequent retrogradation aims at keeping the water-to-starch ratio constant. The merit of such approach is questionable because the overall water content is not kept constant and, subsequently, the availability of water to plasticize the starch polysaccharides is reduced (Farhat et al 1998). Ideally, the water content conditions should be selected to maintain a constant hydration level for the starch. However, this is not easily achievable because the partitioning of water in multicomponent systems depends on the overall water content (Farhat et al 1996, Mousia et al 2000) and temperature, but also on the degree of order of the components of the blend.

This article reports a study of the effect of xylose, fructose, and sucrose on the kinetics of retrogradation of extruded waxy maize starch systems. Limited aspects of the effect of sugars on the isothermal retrogradation has been presented at two conferences (Farhat et al 1997, 1998). However, the main aim of this work is to assess the extent to which the effect of a given sugar on the rate of starch retrogradation depends on the water content of the system and the storage temperature.

MATERIALS AND METHODS

Sample Preparation

Waxy maize starch was supplied by National Starch and Chemical Co., Manchester, UK. Sucrose, fructose, and xylose were obtained from Sigma Chemical Co. Ltd., Poole, UK.

Known amounts of waxy maize starch and sugar were mixed using a Kenwood Peerless planetary mixer for 1 hr and then extruded using a Clextral BC-21 twin-screw corotating extruder.

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The extrusion temperature profile was 40, 110, 120, and 80°C. Distilled water was pumped into the second zone of the barrel to achieve the required water content, taking into account the moisture content of the raw materials. Water contents were determined directly after extrusion by drying the sample in a vacuum oven at 70°C for 24 hr. All sample compositions are reported on a total dry solid basis (%). The samples were sealed and stored at 313 ± 0.1 K in the nuclear magnetic resonance (NMR) spectrometer probe head for isothermal retrogradation studies or in incubators set to a range of temperatures between 277 and 353 ± 1 K for the study of the effect of temperature on the kinetics of starch retrogradation. Moisture content measurements after storage revealed a maximum of 1% water loss from the sealed samples stored at >313 K and $<0.5\%$ for those stored at lower temperatures.

Physical Measurements

X-ray diffraction. Wide angle X-ray diffraction (XRD) is the most widely used technique for the characterization of crystalline order. Its applications in the area of synthetic and natural polymers, particularly in terms of the determination of crystallinity indices, has been extensively reviewed by Alexander (1969).

X-ray diffractograms were recorded for 2θ angles at 4–38° using a diffractometer (PW 1730, Philips Electronics Ltd., UK) equipped with a copper source operating at 40 kV and 50 mA (Cu K_α radiation of 0.154 nm wavelength).

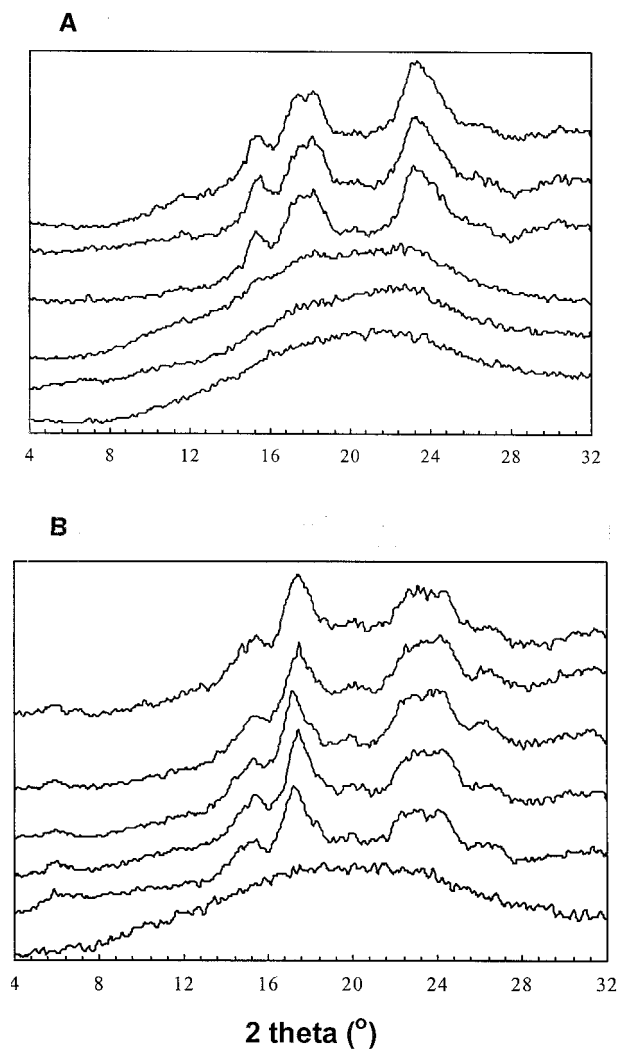


Fig. 1. X-ray diffraction spectra recorded at different storage times for extrudates of waxy maize starch and water (100:35) (A) and waxy maize starch, fructose, and water (90:10:35) (B). Bottom to top: shortly after extrusion, after 1, 2, 3, 4, and 6 days of storage at 295 ± 2 K.

1H relaxation NMR. NMR relaxometry is a well-established technique for the study of molecular mobility in a range of systems including foodstuffs. Several mechanisms govern the relaxation of the 1H net magnetization in the xy plane (M_{xy}), a phenomenon also known as spin-spin relaxation. These include magnetic field dephasing contribution resulting from the presence of other resonant and nonresonant nuclei, chemical shift anisotropy, susceptibility effects, and magnetic field inhomogeneity. The dephasing of a particular spin depends on the neighboring dipoles. This relaxation mechanism is very efficient in solids where mobility is reduced and, thus, the static magnetic component created by neighboring dipoles is large. In more mobile systems such as liquid, the static dipolar fields average out to very small values as a result of random molecular motion leading to less effective dephasing process and long decay times (T_2) for the transverse magnetization (M_{xy}) (Martin et al 1980, McBrierty et al 1981).

Samples were sealed in tubes 8 mm in diameter. NMR measurements were performed using a benchtop Minispec PC120 (Bruker, UK) operating at 313 ± 0.1 K and 20 MHz. The spin-spin relaxation times (T_2) were measured using the Carr-Purcell-Meiboom-Gill (CPMG) 90–180° pulse sequence with an echo time $2\tau = 524 \mu\text{sec}$. One hundred sixty echoes were acquired and the decay was successfully fitted to a single exponential.

Measurements of T_g and T_m

To assess the effect of sucrose on the T_g of amorphous waxy maize starch and the T_m of the recrystallized amylopectin, nonexpanded waxy maize starch-sucrose extrudates containing 0 (control), 10, and 30% sugar, and a moisture level of 32–35% (w/w, dsb) were prepared and sealed as described earlier. The samples were allowed to retrograde for two weeks at 60°C. The extent of retrogradation was assessed using XRD. A range of water contents was subsequently achieved by removing the samples from the airtight bags and storage at 5°C over saturated salt solutions (desorption). Relative humidities at 30–98% were used to achieve moisture contents at 10–50% (w/w, dsb). For higher water contents, a known amount of water was added directly to the sample in the differential scanning calorimetry (DSC) aluminum pan before sealing the pan and the sample was left overnight to equilibrate before subsequent analysis.

Calorimetric experiments were performed (DSC-7, Perkin-Elmer, UK) at a heating rate of 10°C/min. An empty pan was used as a reference. The T_m was defined as the onset of the melting peak measured on the first DSC scan, while the T_g was determined from the midpoint of the glass transition event recorded on the second scan of the same sample.

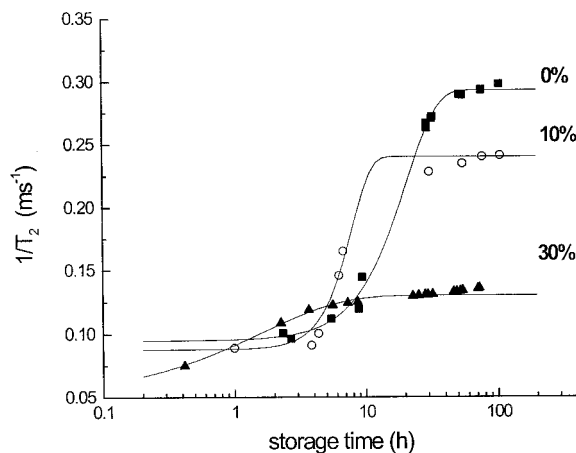


Fig. 2. Starch retrogradation at 313 K monitored by the change in spin-spin relaxation rates ($1/T_2$) for different concentrations of fructose in samples containing $35 \pm 1\%$ water (w/w, dsb). Lines represent nonlinear fit using Equation 1.

RESULTS AND DISCUSSION

For all samples investigated, the XRD recorded shortly after extrusion showed a single broad peak centered at $2\theta \approx 20^\circ$, which is typical of amorphous fully gelatinized starch. It is, however, very likely that the degree of starch conversion (i.e., the extent of degradation that the amylopectin macromolecules have experienced during the extrusion process) will vary with sample formulation. Indeed, as shown by Sriburi et al (1999), the loss of molecular order measured by XRD and birefringence constitute only the first step in the transformation of starch during processing and, consequently, other techniques such as water absorption and water solubility indices, molecular weight determination, viscosity, etc., are required to probe the molecular degradation of the starch polysaccharides. Fan et al (1996) clearly demonstrated the role of plasticizers such as water and sugars in decreasing the degree of starch conversion during a high shear process such as extrusion. It is therefore anticipated that the extent of amylopectin degradation is the highest for lowest water content control sample and the lowest for highest water content samples containing fructose and xylose.

No evidence of sugar crystallization was observed from XRD results, with the exception of some samples containing 30% xylose and <22% water, which showed intense sharp peaks characteristic of crystalline sugars (line width $<0.5^\circ$). These samples were not considered for this study due to a highly phase-separated character.

The X-ray spectra recorded at different storage times indicated an increase in the degree of crystallinity of the starch as the retrogradation process progressed (Fig. 1). While a typical A-type XRD diffractogram was obtained for the retrograded waxy maize starch and water system (Fig. 1A), fructose enhanced the formation of

the pseudo-B crystalline form (Fig. 1B). This polymorph shows all the characteristic features of the B-type X-ray pattern such as the sharp peak at 17.2° (this peak is doubled by a second peak at $\approx 18.2^\circ$ in the A-type) and the presence of two peaks at ≈ 22.3 and 24.2° (these two peaks merge to one single peak at $\approx 23.2^\circ$ in the A-type), but the peak at 5.6° (1.6 nm spacing), which is very pronounced for the B-type polymorph, was much smaller. The effect of fructose, sucrose, and xylose on the polymorph formed during the retrogradation of extruded waxy maize starch, which is initially of the A-type, has been discussed elsewhere (Farhat et al

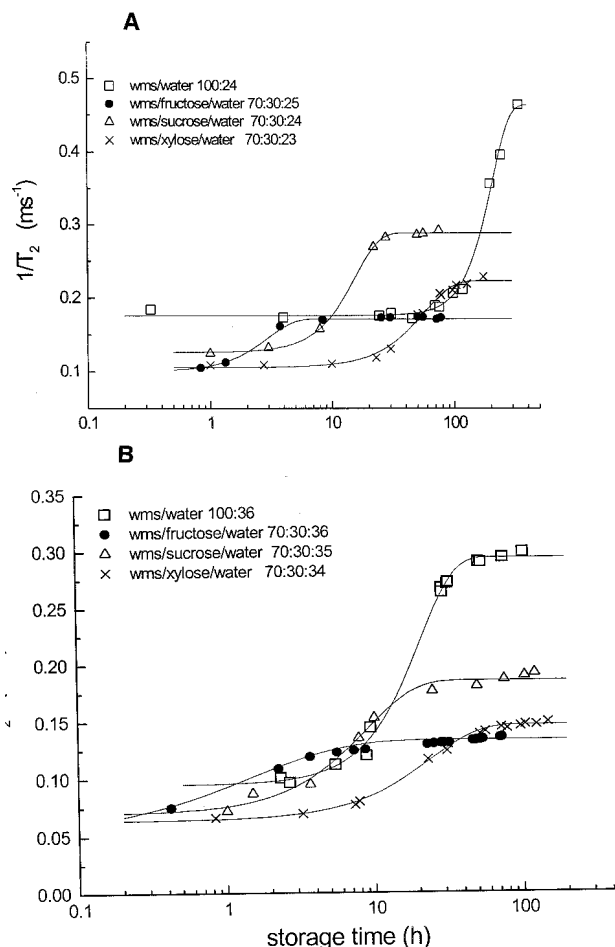


Fig. 3. Changes in spin-spin relaxation rates ($1/T_2$) with storage time (at 313 K) for samples containing 30% fructose, sucrose, or xylose. Water content: **A**, $24 \pm 1\%$; **B**, $35 \pm 1\%$.

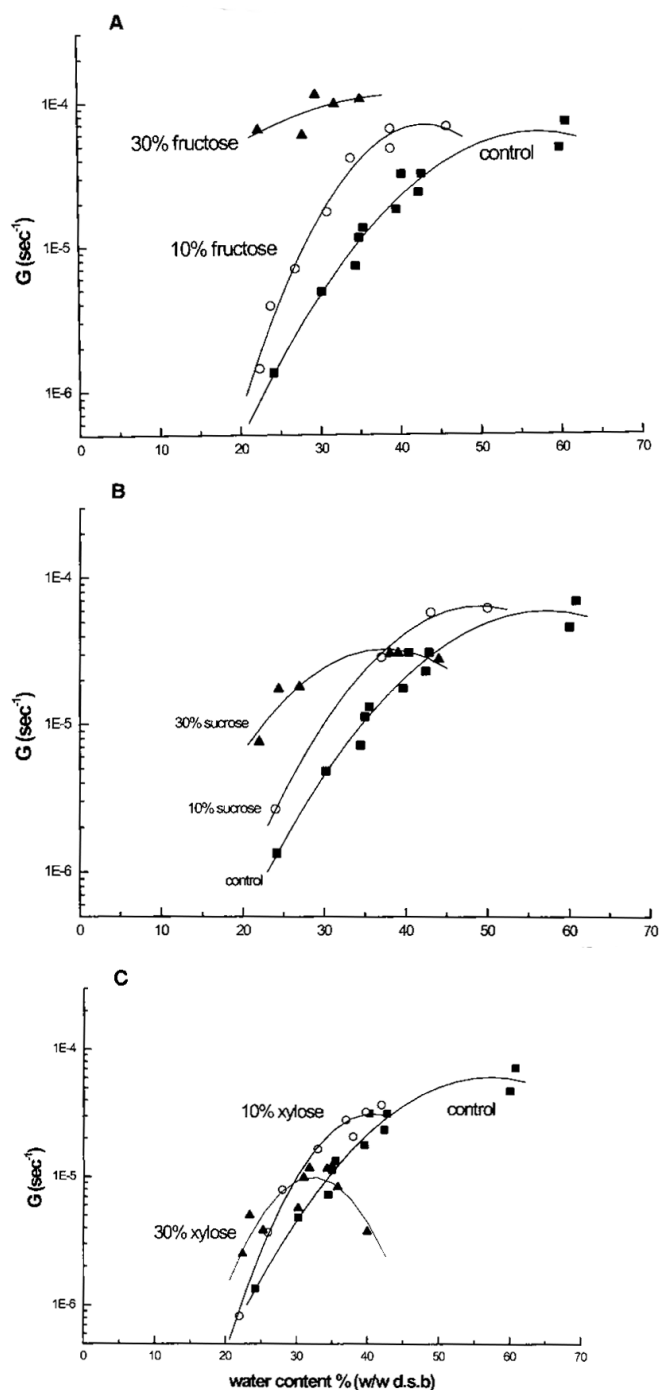


Fig. 4. Effect of water content on the retrogradation rate (G) at 313 K derived by fitting Equation 1 to the nuclear magnetic resonance relaxation rates: **A**, waxy maize starch and fructose; **B**, waxy maize starch and sucrose; **C**, waxy maize starch and xylose.

1998) where, based on XRD, ^{13}C solid-state NMR and DSC observations reported by Wang and Jane (1994), the inclusion of sugar molecules within the starch crystallites was proposed. Three arguments in support of the possible incorporation of sugar molecules in the amylopectin crystalline structure were presented. First, at water contents where an A-type organization would be expected, in the presence of sugar, a so-called pseudo-B type X-ray pattern was observed. The absence of the $5.8^\circ 2\theta$ peak in the XRD spectrum distinguishes the spectrum of the pseudo-B from that typically recorded on B-type polymorphs. Second, at 298 K and a relatively high water content (44%, dsb), sugar would be expected to be highly mobile (Farhat et al 1996) and not detectable by the ^{13}C solid-state CP-MAS NMR experiment which is designed to examine rigid solid-like components. A fraction of the sugar NMR signal was present in the CP-MAS spectrum of a retrograded waxy maize starch, sucrose, and water system (70:30:44). Finally, Wang and Jane (1994), who studied the effects of various sugars on starch retrogradation detected a change in T_g' with storage for the samples containing sugar, while no change was observed for the control. A plot of the T_g' versus the extent of retrogradation (both measured by DSC) yields straight lines with positive gradients for the samples containing fructose and sucrose compared with a zero gradient for the control sample and for the sample containing the larger molecular weight additive, maltodextrin. Their interpretation was that some sugar was absorbed or included in the retrograded starch phase.

As reported elsewhere (Farhat 1996; Farhat et al, *in press*) for waxy maize starch and water systems, the ^1H NMR spin-spin relaxation rate ($1/T_2$) measured using the spin-echo (CPMG) pulse sequence decreased as the retrogradation progressed with increasing storage time. In this study, the spin-spin relaxation rates were used to probe the extent of retrogradation and characterize the kinetics because the technique offered many practical conveniences such as limiting the loss or uptake of moisture during the measurement and reliable temperature control (± 0.1 K) that were difficult to implement on the XRD equipment. The advantages of using ^1H NMR relaxometry over other techniques such as XRD, DSC, etc. are discussed elsewhere (Farhat et al, *in press*).

The rate of retrogradation was calculated by fitting an Avrami-like equation to the $1/T_2$ results using nonlinear χ^2 -minimization routine (Farhat 1996; Farhat et al, *in press*):

$$U(t) = \exp[-(Gt)^n] \quad (1)$$

where $U(t)$ is the uncrystallized amylopectin fraction at time t , G is the rate of retrogradation, and n is an exponent.

Effect of Added Sugars on Isothermal Retrogradation

The isothermal retrogradation rates of waxy maize starch and water systems of various concentrations reported elsewhere (Farhat et al, *in press*) provided a control allowing the assessment of the effect of each sugar on the kinetics of starch recrystallization retrogradation. Sugars affected the behavior of the various NMR parameters such as the relaxation rates versus storage time diagrams. These effects could be divided into three categories that are directly or indirectly related: 1) the sugars plasticize the amorphous gelatinized waxy maize starch and increase its molecular mobility and T_2 ; 2) the sugars affect the kinetics of the starch retrogradation and the rate at which the rigid ordered component of the starch with a short T_2 , is formed; and 3) the replacement of a fraction of amylopectin by sugar introduces a new population of hydrogen bonding and chemical exchange partner of higher mobility than that of the polymer matrix, increasing the spin-spin relaxation time of water relative to that in the systems containing no sugar.

The effect of sugars on the evolution of the NMR relaxation rate with storage time through starch retrogradation depended strongly on the type and concentration of sugar in the system: the incorporation of fructose in waxy maize starch extrudates containing $35 \pm 1\%$ water stored at 313 K increased the rate of retrogradation (Fig. 2). The increase was proportional to the amount of fructose in

the system: at a level of 10% (w/w, dry solid) the retrogradation reached its equilibrium value after ≈ 12 hr compared with 4 hr for a fructose concentration of 30% and ≈ 50 hr for the control system.

The comparative effects of the inclusion of 30% fructose, sucrose, or xylose in the same storage conditions (313 K) at two water contents is illustrated in Fig. 3. At $24 \pm 1\%$ water content, all three sugars enhanced the rate of isothermal retrogradation. Their effect followed the order: xylose < sucrose < fructose. At $35 \pm 1\%$ water content, xylose delayed the retrogradation. Indeed, $1/T_2$ reached its plateau value after ≈ 73 hr compared with 50 hr for the control. The role of water content in the effect of sugars on the retrogradation was investigated by studying samples with a range of water contents.

It is worth noting that sugar considerably decreased the values of the initial (zero storage time, $t = 0$) and the equilibrium (long storage time $t = \infty$) relaxation rates, and the magnitude of the decrease was proportional to both the amount of sugar (Fig. 2) and the molecular weight of the sugar (Fig. 3). This is believed to be the result of the relationship between the molecular mobility and the average molecular weight of the combined starch and sugar component acting like a relaxation sink through which the spin-spin relaxation of water by chemical exchange occurs. In this terminology, the sugars have a higher molecular mobility than amylopectin and therefore constitute a less efficient relaxation sink for the water molecules. A detailed discussion of the mech-

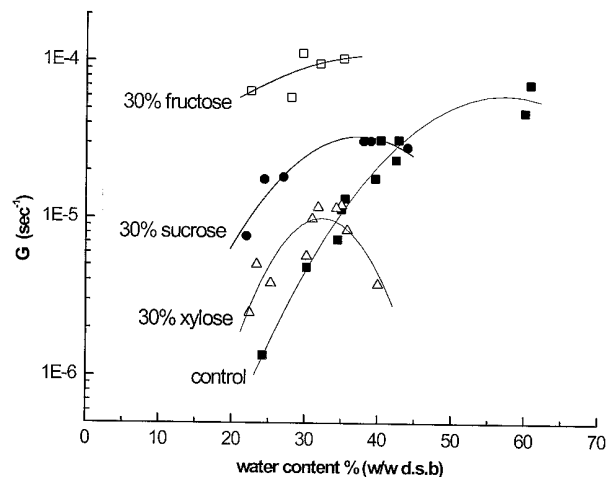


Fig. 5. Effect of type of sugar on the dependence of the retrogradation rate (G) at 313 K for starch in waxy maize starch and sugar (70:30) extrudates on water content.

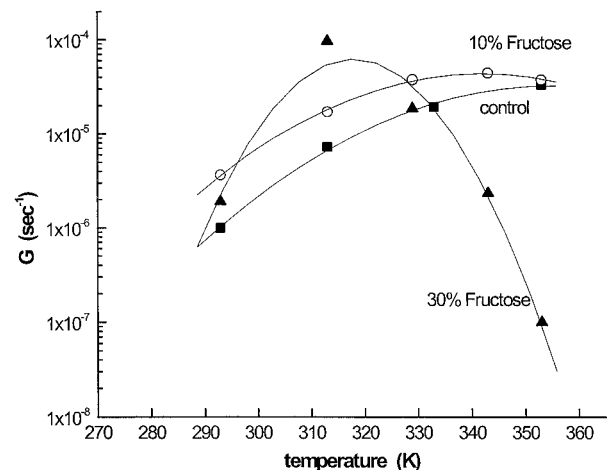


Fig. 6. Effect of storage temperature on the retrogradation rate (G) for waxy maize starch and fructose (100:0, 90:10, and 70:30) containing 32% water (w/w, dsb).

anisms of relaxation of water in biopolymer systems is outside the scope of this article. Belton et al (1987, 1988) provided a comprehensive series of articles and a review (Belton 1994) on this topic.

Role of Water Content

The effect of water content on the rate of isothermal retrogradation of extruded starch and water systems has been successfully described elsewhere (Farhat et al, *in press*) by adapting the Lauritzen-Hoffman theory of crystallization of chain-folded polymers. The effect of water on the T_g and T_m of starch was calculated using the Ten Brinke et al (1983) and the Flory(1953) equations, respectively. A similar quantitative approach was not attempted in this study due to the higher numbers of unknown parameters that needed to be calculated due to the extra component, sugar. Such calculations would carry more uncertainties in this case due to the limited number of experimental observations relative to the number

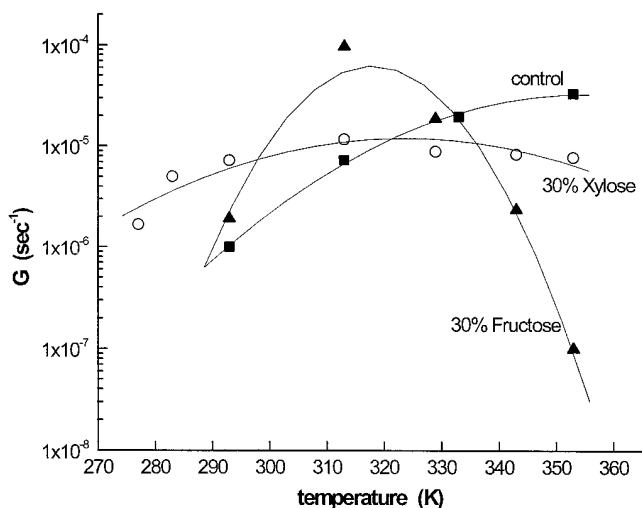


Fig. 7. Effect of storage temperature on the retrogradation rate (G) for waxy maize starch and fructose and waxy maize starch and xylose (70:30) containing 32% water (w/w, dsb).

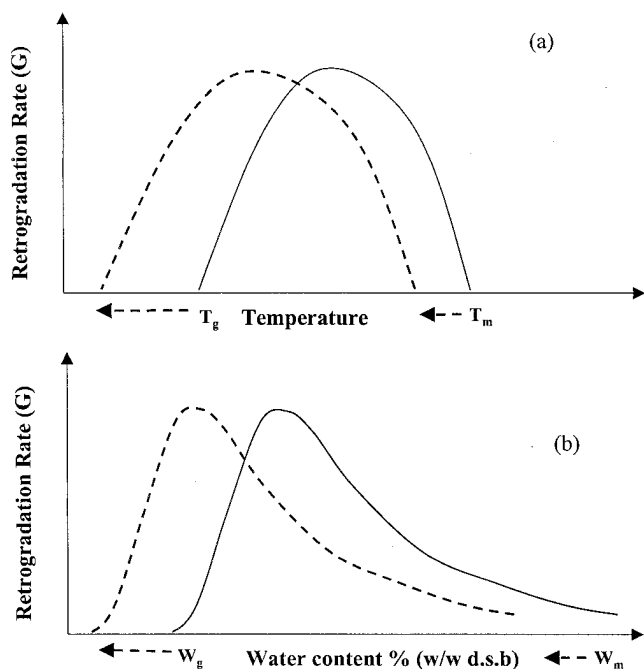


Fig. 8. Effect of sugars on the retrogradation rate (G) vs. storage temperature (a) or water content (b). Solid line = control; dotted line = sugar. Shape of the diagrams in (b) is based on the modeling of experimental waxy maize starch and water data (Farhat et al, *in press*).

of unknown parameters to be numerically adjusted. Consequently, only a qualitative interpretation, based on the same approach will be attempted here.

While fructose increased the rate of isothermal retrogradation over the entire range of water contents investigated, and the effect at a particular water content was proportional to the amount of sugar (Fig. 4A), sucrose and xylose showed more complex patterns (Fig. 4B and C). At a sugar concentration of 10%, both sucrose and xylose enhanced the isothermal retrogradation rate, and the magnitude of the effects was comparable for the three sugars investigated. At 30%, xylose accelerated the process at water contents <35% (w/w, dsb) but decreased the rate constant when more water was present; this crossover took place at a moisture content of $\approx 42\%$ for sucrose (Fig. 5).

Storage Temperature

The retrogradation of waxy maize starch and water and waxy maize starch, sugar, and water extrudates (fructose and xylose) was studied at 277–353 K for a water content of $32 \pm 1\%$ (w/w, dsb). In general, the results showed part of the expected bell-shaped pattern typical of the dependence of the crystallization kinetics of many polymers and biomolecules such as amylopectin on the storage temperature (Farhat et al, *in press*). While the sample containing 10% fructose showed enhanced retrogradation rate compared with the control sample over the entire storage temperature range, fructose at 30% enhanced the retrogradation at storage <330 K and had the opposite effect at higher temperatures (Fig. 6). For

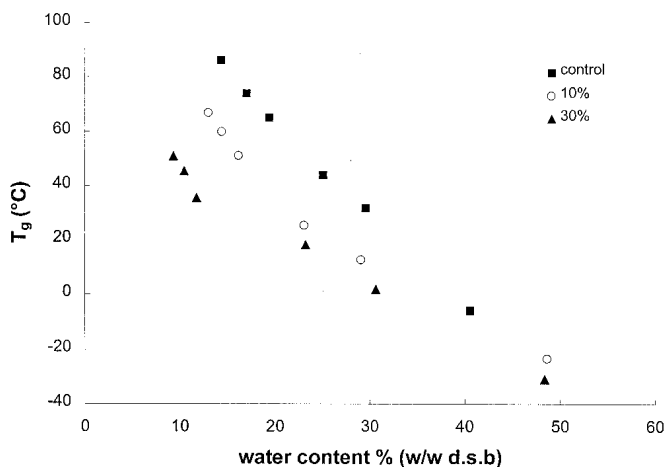


Fig. 9. Effect of sucrose content on the glass-transition temperature (T_g) (differential scanning calorimetry midpoint) of extruded waxy maize starch and sucrose mixtures (100:0 [control], 90:10, and 70:30).

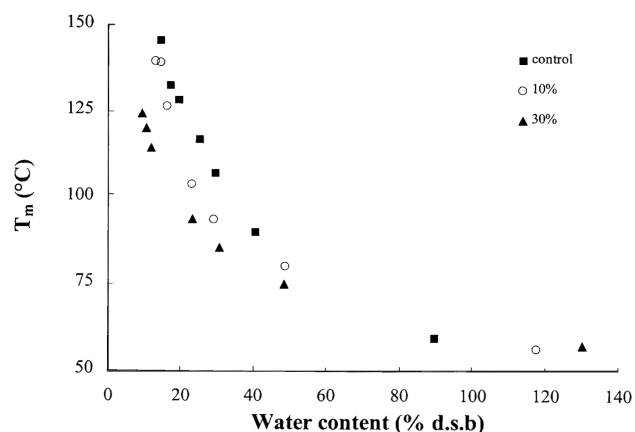


Fig. 10. Effect of sucrose content on the melting temperature (T_m) (differential scanning calorimetry onset) of retrograded waxy maize starch and sucrose extrudates (100:0 [control], 90:10, and 70:30).

waxy maize starch and xylose (70:30), this crossover in the rate versus temperature diagrams occurred at a temperature ≈ 10 K below that observed for the corresponding waxy maize starch and fructose system (Fig. 7).

This behavior is very similar to that observed in the study of the effect of water content on the rate of isothermal retrogradation which could be schematically represented in a generalized model (Fig. 8). The shift of the T_g to lower temperatures and subsequently W_g , the water content at which the T_g equals the storage temperature is expected on the basis of the role of sugars in plasticizing amylopectin (Kalishevsky et al 1993). However, the decrease in the T_m of recrystallized amylopectin and subsequently W_m , the extrapolated temperature at which the retrograded crystallites melt at the storage temperature is, in the first order, counterintuitive. Indeed, several workers have reported the role of sugars in increasing the melting temperature of starch crystallites (Lelievre 1976, Kim et al 1992). It is, however, important to note that the behavior of native granular starch and of recrystallized starch may be different due to the importance of the diffusion of water from the amorphous background to the crystalline lamellae for the melting of native starch (Jenkins 1994). The most likely reasons for this decrease in T_m , however, are 1) the possible presence of sugar molecules within the starch crystalline regions reflected by the observation of the pseudo-B XRD pattern, or 2) plasticization of the amorphous regions leading to a decrease of T_m as described by Slade and Levine (1991) based on the fringed micelle model for partially crystalline polymers. It is believed that the pseudo-B form involving the sugar inclusion can only occur in the starch and sugar systems if they are retrograded at temperatures below $\approx 30^\circ\text{C}$ at 20% water content (w/w, dsb) and 60°C at 50% water content. While in this temperature region, the mechanism by which sugar inclusion in the starch crystals can be invoked. At higher temperatures, the plasticization theory put forward by Slade and Levine (1991) must be the dominant process.

The value of G_{\max} , the maximum of the rate versus temperature or rate versus water content diagrams may depend on the type and concentration of sugar present in the system.

The T_g obtained from the second DSC scan showed a clear plasticization of the amorphous waxy maize starch by sucrose (Fig. 9). The effect is less pronounced at higher water contents. Such behavior is in agreement with the findings of Kalichevsky et al (1993) on comparable waxy maize starch and sugar systems. A similar behavior was found for the melting temperature of the amylopectin crystallites formed during the retrogradation of the amorphous waxy maize starch (Fig. 10). The XRD spectra recorded on the waxy maize starch and sucrose samples retrograded at 60°C for the DSC study were of the A-type regardless of the sugar content. This is in agreement with the effect of storage temperature on the type of crystallization. Indeed, at 32–35% water content, the moisture content of the samples, amylopectin is expected to crystallize in the A-form for storage >40 – 45°C and therefore the decrease in T_m observed in Fig. 10 is mainly the consequence of the plasticization of the amorphous regions of amylopectin by sucrose.

The hypothesis suggested in Fig. 8 might provide a generalized interpretation to the contradictory reports on the effect of sugars on the kinetics of starch retrogradation. It is suggested that these contradictions result from the fact that the role of a particular sugar depends greatly on its concentration in the system, the water content, and the temperature of retrogradation. For example, the results of Marsh (1986) suggesting that xylose inhibited the retrogradation of wheat starch gels while sucrose delayed it and fructose enhanced it are in agreement with the findings of this study. The systems investigated by Marsh contained 33 and 48% water (wb) (50–92%, db). Furthermore, I'Anson et al (1990) findings regarding sucrose inhibiting entirely the retrogradation of wheat starch gels can also be explained considering the sucrose content (50%, db) and the water content (50% db) of their study on the basis of the hypothesis proposed in this study.

CONCLUSIONS

The effect of three sugars (fructose, sucrose, and xylose) on the rate of retrogradation of waxy maize starch extrudates over a range of water contents and storage temperatures was studied. The role of sugar depended to a great extent on its type and concentration, but more importantly on the water content of the system and the storage temperature at which the retrogradation process occurred. The results could be rationalized in the terms of the effect of sugars on the T_g of the amorphous gelatinized starch and the T_m of the crystallites formed by retrogradation of this gelatinized starch in the presence of sugars.

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