Journal of Cereal Science **27** (1998) 147–155

Effects of Starch Gelatinisation on the Thermal, Dielectric and Rheological Properties of Extruded Corn Masa

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Received 3 June 1996

ABSTRACT

The effects of processing on starch gelatinisation of corn masa prepared by extrusion were monitored by observing the changes in thermal diffusivity, starch crystallinity, peak viscosity and the differential dissipation factor (DDF). The results show that there is a threshold processing temperature (Tc), about 80°C, above and below which different behaviour patterns for the measured properties are observed. The thermal diffusivity, starch crystallinity and the position of a peak in the DDF have a maximum at approximately Tc = 80°C. The peak viscosity decreases monotonically for Tc up to 80°C and then remains constant. On the bases of textural characteristics of masa and tortilla, this work indicates that the degree of starch gelatinisation in the extruded masa is optimum at temperatures at approximately 80°C where the room temperature thermal diffusivity and differential dissipation factors have maximum values. Textural analysis made in tortillas with masas prepared at various temperatures support the above conclusions.

Keywords: crystallinity, thermal diffusivity, starch gelatinisation, corn masa.

INTRODUCTION

At the present time the only process to produce tortillas in a commercial scale is the traditional nixtamalisation process. The reason is that other methods have not been able to produce tortillas with a quality comparable or better than that of the traditional process. This work presents results that indicate that the extrusion method is a viable candidate to produce tortillas with the proper quality.

Starch gelatinisation has been studied by methods based on the observation of changes in various properties of starch granules when they are heated in aqueous solution. Starch gelatinisation is characterised by several changes in its granular structure, such as swelling of granules, exudation of some complex molecules, dissolution of some granules, loss of the degree of crystallinity, etc. The processing temperature must allow sufficient disruption of the granules to generate these structural changes. Several techniques have been used to characterise the starch gelatinisation process,

ABBREVIATIONS USED: α = thermal diffusivity; DDF = differential dissipation factor; Tc = processing temperature; PA = photoacoustic; C% = crystallinity; RVU = Relative Viscosity Units (1 RVU = 10 cp).

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0733–5210/98/020147 + 09 $25.00/0/jc970162

however, the understanding of this process is still incomplete. The methods used for the determination of starch gelatinisation are based on observations of the micro and/or macroscopic changes in various properties of the starch. Some of the most commonly used are: observation of swelling by microscopy[1,2], staining with dyes[3], birefringence loss in polarised light[4], differential scanning calorimetry[5], X-ray diffraction[6], electrical conductivity[7] and nuclear magnetic resonance[8].

Recently the photoacoustic (PA) technique has been used to characterise the thermal properties of instant corn dry masa flour and baked tortillas[9–11]. The PA method can be used to observe changes in thermal parameters such as thermal diffusivity, thermal conductivity and specific heat. These parameters are extremely sensitive to changes in the chemical composition and structure of starch and are affected by the degree of gelatinisation during processing. On the other hand, the dielectric method based on the changes of DDF has the potential to be used for measuring the structural changes in starch-derived foods during processing. This paper discusses the use of PA, X-ray diffraction and dielectric measurements to monitor the gelatinisation of corn starch during its processing by extrusion. Also the textural properties of tortillas produced from masa with different degrees of gelatinisation were evaluated. There is a strong dependence of these properties on the processing temperature (Tc). The room temperature thermal diffusivity and the position of the low frequency peak in the DDF (measured as a function of Tc) shows a maximum at about 80 °C. This temperature is determined as the optimum for preparing masa for tortilla production. Aiming for a better understanding of the changes induced by the processing temperature, the thermal and dielectric data are further supplemented by viscosity and X-ray diffraction.

EXPERIMENTAL

Sample preparation

The continuous extrusion process used to obtain fresh corn masa has previously been reported[12]. Whole corn powder, water and lime ($Ca(OH)_2$) were used as the starting materials. The whole corn powder was obtained using a hammer mill with a 0·8 mm sieve. The starting mixture, which included for each kilogram of corn powder 0·73 L of water and 2·5 g of lime, was fed into the extruder for processing. Samples of extruded masa were prepared at various barrel temperatures (Tc) from 50 °C to 100 °C, the residence time was kept constant at 1·3 min for all runs. Corn masa was also obtained using the traditional thermo-alkaline method – whole corn grains were cooked in water and lime at boiling temperature for about 45 min – the cooked grains were then soaked in the same cooking water for approximately 10 h to achieve equilibrium moisture, and ground to obtain masa with the proper consistency.

Apparent viscosity

A Rapid Visco Analyzer (RVA) (Newport Scientific Pty, Sydney, Australia) was used to measure the apparent viscosity of samples as a function of temperature. The procedure consisted of following steps: the sample moisture was determined (41–46% w.b.) in all cases. A sample of masa that weighed 3·0 g (adjusted to 14% m.b.) was used. Distilled water was then added to keep the total weight of water and masa constant at 28 g. The time–temperature sequence used was as follows: the sample is initially held, with the paddles rotating, at 50 °C for 2 min to stabilise the temperature and ensure uniform dispersion and wetting, then it is heated up to 92 °C at a constant heating rate of 5·6 °C per min, held at this temperature for 5 min and then cooled down to 50 °C in 7·5 min at the same rate. The viscosity was obtained in RVU (1 RVU = 10 cp).

X-ray diffraction

The X-ray diffraction patterns of the samples prepared at different Tc were taken in a Phillips diffractometer operating at 35 kV, with CuKα radiation. The diffractograms were recorded from 2 to 60 in the 2θ scale. At higher angles no significant diffraction intensity was measured. Powders of dehydrated masa samples were densely packed in an aluminium frame for measurement. The crystallinity (C%) was calculated by normalising the integrated diffracted intensity to the integrated non-coherent intensity. The non-coherent intensity was obtained by subtracting the sharp diffraction peaks from the total diffraction pattern.

Dielectric measurements

Dielectric measurements were made using a differential method that determined the dielectric

properties at two different time intervals. A Schulmberg model SI 1260 Impedance/Gain-Phase Analyzer operating at 200 preprogrammed frequencies between 1 and 30 Hz, with a voltage amplitude of 3 volts, was used. Samples were placed in the sample holder that consists of two parallel stainless steel circular plates 15 cm in diameter and 7 mm apart. Electrical connections were attached to each plate and data were recorded at room temperature. In order to eliminate possible stray admittance measurements from the sample holder, a built-in nulling feature was employed to determine the admittance data. Taking advantage of this property, we took the admittance values as a function of frequency using two different time measuring conditions. This allowed us to determine the differential admittance conditions at two different time intervals. The detailed procedure for measurements of the dissipation factor for each frequency interval will be described elsewhere. The DDF (loss tangent) is defined as the ratio of the imaginary to the real parts of the dielectric function of the material, in other words it is the ratio of the dissipated to the stored electrical energy in the system.

Thermal diffusivity measurements

The thermal diffusivity of fresh masa samples prepared at different Tc was measured using the PA technique. All measurements were performed at room temperature. The experimental set-up is schematically shown in Figure 1. It consists of a 150 W tungsten filament lamp. The polychromatic beam from the lamp is mechanically chopped and focused onto the sample. The sample plays the role of a second window enclosing the PA cell. The disc-shaped sample is supported in an acrylic ring 440 μm thick with a 9 mm diameter role. One side of this hole is closed using a 50 μm thick piece of Al foil glued to the acrylic ring. The rear-side of this Al foil is in contact with the outside air and exposed to the rear-side light beam illumination. The sample-supporting acrylic ring is fixed to the cell body with silicon grease. A commercial electret microphone mounted in one of the cell walls is in contact with the air inside the PA chamber. The signal from the microphone is connected to a lock-in amplifier and recorded as a function of the modulation frequency. This arrangement corresponds to a heat transmission configuration. That is, the heat deposited at the rear-side face of the sample (due to the light-into-heat conversion at the Al foil) first diffuses through it before reaching the PA air chamber where it causes pressure fluctuation detected by the microphone. By monitoring the PA signal as a function of the light chopping frequency, the thermal diffusivity of the sample can be obtained[13].

For the rear-side illumination configuration, schematically shown in Figure 1, the thermal diffusion model of Rosencwaig and Gersho[14] predicts the pressure fluctuation δP in the air chamber as given by[15]:

$$\delta P=\frac{\gamma P_0 I_0(\alpha_g\alpha_S)^{1/2}\exp j(\omega t-\pi/2)}{2\pi l_g T_o k_s f\,\mathrm{Sinh}(l_s\sigma_s)} \qquad (1)$$

where γ is the air specific heat ratio, $P_o(T_o)$ is the ambient pressure (temperature), f is the modulation frequency and l_i, k_i and α_i are the length, thermal conductivity and thermal diffusivity of material i, respectively. Here the subscript i denotes the sample (s) and the air (g), respectively, and $\sigma_i=(l+j)\,a_i$; $a_i=(\pi f/\alpha_i)^{1/2}$, is the complex thermal diffusion coefficient of material i and $j=\sqrt{-1}$. In arriving to Equation (1) we have assumed that the sample is optically opaque and that the heat flux into the surrounding air is negligible.

The implicit optical opaqueness was ensured by the use of the thin absorbing Al foil attached to the sample, as described previously. For a thermally thin sample, namely $l_s a_s \ll 1$, Equation (1) reduces to

$$\delta P\approx\frac{\gamma P_o I_o\alpha_s\exp[j(\omega t-3\pi/4)]}{(2\pi)^{3/2}l_g T_o l_s k_s f^{3/2}}. \qquad (2)$$

In other words, the PA signal amplitude decreases as $f^{3/2}$ as one increases the modulation frequency. In contrast, if the sample is thermally thick (i.e. $l_s a_s \gg 1$) one obtains from Equation (1).

$$\delta P\cong\left(\frac{\pi P_0 I_0(\alpha_g\alpha_s)\exp[-l_s(\pi f/\alpha_s)^{1/2}]}{\pi l_g T_o k_s f}\right)\times \qquad (3)$$

$$\exp[i(\omega t-\pi/2-l_S a_S)]$$

Equation (3) implies that, for a thermally thick sample, the amplitude of the PA signal decreases exponentially with the modulation frequency according to

$$\delta P=A\exp(-b\sqrt{f}/f). \qquad (4)$$

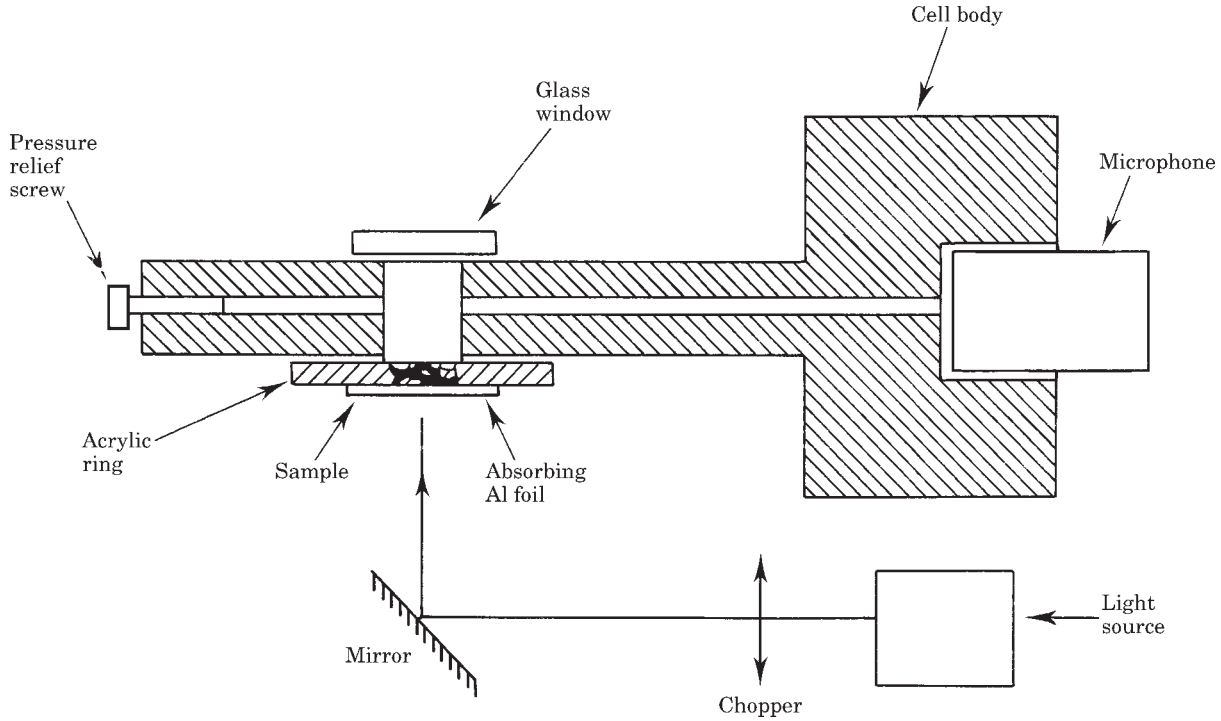

Figure 1 Schematic arrangement for room temperature PA measurements. The sample fills the 9 mm diameter, 450 μm thick acrylic ring.

The thermal diffusivity α_s can then be obtained from the right light-modulation frequency behaviour of either the signal amplitude or its phase. For the signal amplitude, α_s is obtained from the experimental data fitting from the coefficient b in the argument of the exponential $(-b\sqrt{f})$. However, for a plate-shaped sample, the contribution to the PA signal from the thermoelastic bending of the sample cannot be neglected, especially for thermally thick samples, as has been demonstrated by Rousset and Lepoutre[16,17]. This effect is essentially due to the temperature gradient inside the sample along the z-axis. Owing to the existence of this temperature gradient parallel to the z-axis, thermal expansion depends on z. This z-dependence of the displacement along the radial direction induces a bending of the plate in the z-direction (drum effect), i.e. the vibrating sample acts as a mechanical piston, thereby contributing to the PA signal. In the thermally thick regimen, the pressure fluctuation in the air chamber of the PA cell resulting from the thermoelastic properties of the sample[17] is given by

$$\delta P=\frac{3\alpha_T R_0^4 I_0 \alpha_s}{4\pi R_c^2 l_g k_s f}\left[\left(1-\frac{1}{x}\right)+\frac{1}{x^2}\right]^{1/2} \times \exp j[\omega t+\pi/2+\varphi] \quad (5)$$

where $x=l_s a=l_s(\pi f/\alpha_s)^{1/2}$ tan $\varphi-1/x$-1), α_T is the sample thermal expansion coefficient, R_0 the radius of the front hole of the microphone and R_c the radius of the front air chamber. Equation (5) implies that the thermoelastic contribution, at high modulation frequency such that $x\gg1$, δ, P and its phase ϕ approaches 90° as

$$\varphi=\varphi_0+\arctan\left[\frac{1}{(x-1)}\right] \quad (6)$$

Thus for a thermally thick sample, its thermoelastic contribution is dominant, the thermal diffusivity can be evaluated from the modulation-frequency dependence of either the signal amplitude [Eqn. (5)] or its phase [Eqn. (6)].

Firmness of masa and tortillas

The TA.XT2 Texture Analyzer (Texture Technologies Corp., Scarsdale, NY/Stable Micro System, Godalming, Surrey, U.K.) was used to determine adhesiveness and cohesiveness of masa

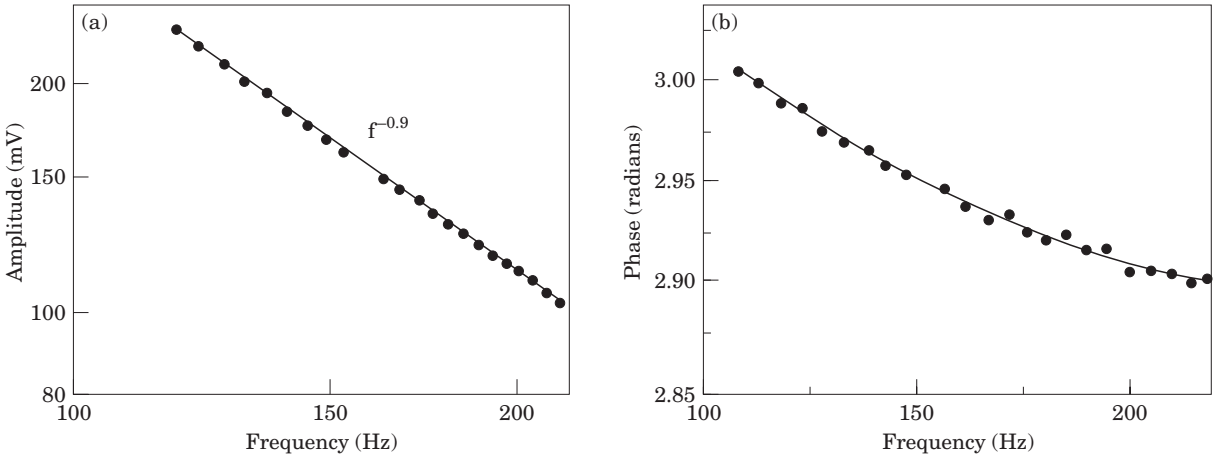

Figure 2 (a) Dependence of PA signal amplitude with the modulation frequency of the incident light, for corn masa processed at a Tc = 50 °C. (b) Dependence of PA signal phase on the chopping frequency for the masa processed at Tc = 50 °C.

and tensile strength and the force required to cut a tortilla. Masa samples (50 g) were shaped in the disc form of $4 \pm 0{\cdot}1$ cm diameter and $1{\cdot}5 \pm 0{\cdot}1$ cm thickness and placed on the metallic platform. The TA-18 probe was attached to the head of the Texture Analyzer and tested for adhesiveness and cohesiveness. Testing conditions were a speed of 2 mm/sec, and a penetration of 4 mm deep. The peak force (g_f) was recorded. For tensile strength and cutting force, a sample consisting of a $3{\cdot}7 \times 9$ cm strip of the middle part of a tortilla was placed on the TA-96 probe, attached to the head of the Texture Analyzer and tested. The texturemeter head moved the probe upwards at 2 mm/sec until the tortilla broke. Two tortillas of each test group were evaluated after 1 h and 24 h (stored inside polyethylene bags) at room temperature (25 °C). The same strips of tortilla were placed on the platform and the TA-90 attachment was used to determine the force required to cut them. The texturemeter head moved the probe downward at 2 mm/sec until the tortilla was cut. The tensile strength and cutting force were expressed as peak force (g_f or kg_f) required to break and cut the strip.

RESULTS AND DISCUSSION

Figure 2(a) shows the dependence of the PA signal amplitude with the modulation frequency of the incident light for a masa sample prepared at Tc = 50 °C. In the frequency range of 100 to 220 Hz the signal amplitude behaves essentially as f^{-1}. This dependence of the PA signal of a thermally thick sample indicates that in this frequency range, the thermoelastic bending is the mechanism dominating the acoustic signal and was the dominant mechanism in all samples. Thus, Equation (5) can be used to determine the thermal diffusivity. The thermal diffusivity can also be obtained from the PA signal phase from Equation (6). We used the latter, and the room temperature thermal diffusivity was obtained from plots similar to those in Figure 2(b), which corresponds to a sample prepared at 50 °C. The values of the thermal diffusivity obtained for masa samples prepared at different Tc are shown in Figure 6(a). Figure 3 shows the X-ray diffraction patterns for dehydrated corn masa samples prepared at different Tc, as well as for the raw corn powder. According to X-ray diffraction data, the structure of starch, can be grouped into four types; A, B, C and V[18]. In Figure 3, the X-ray diffraction trace of the raw maize sample shows sharp diffraction peaks around 5.8, 5.1 and 3.8 Å which corresponds to an A-pattern. From data in this figure, it is evident that in the processed samples, a diffraction peak at about 4·4 Å develops and increases in relative intensity as Tc is increased. The intensity of this latter peak is the strongest in the sample prepared of Tc = 100 °C. This peak is the distinguishing feature of V type starch. Another feature of these V patterns is a diffraction line corresponding to a spacing of about 12 Å. However, the line at 4·4 Å often appears as a first indication of V type complex. The 12 Å line has been observed before on starch processed under selected treatments that might include combinations of moisture, temperature and treatment times[18]. Besides the trans-

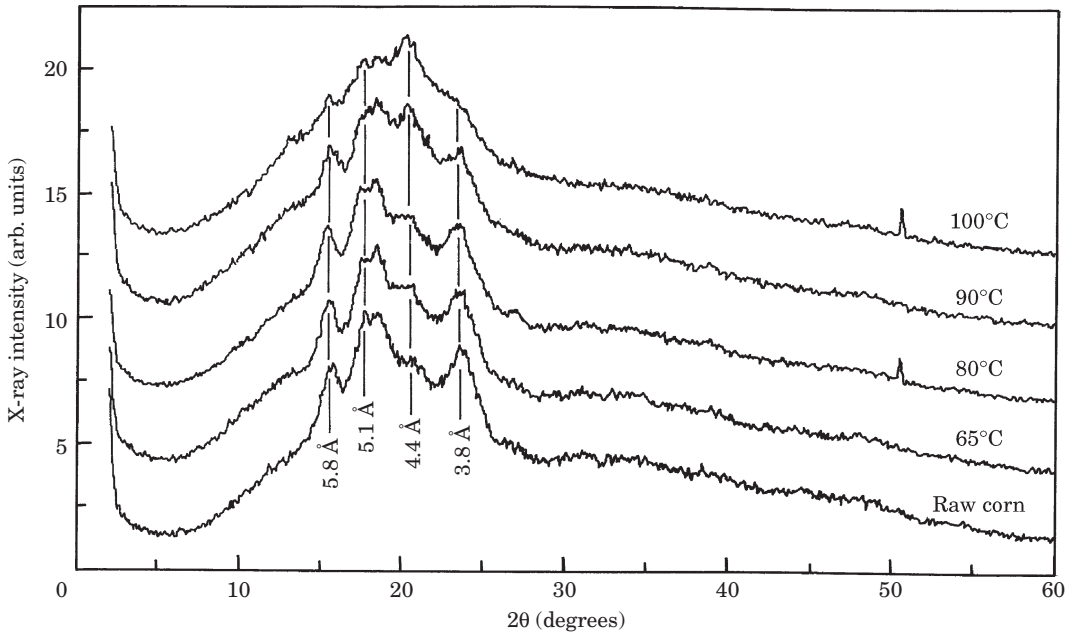

Figure 3 X-ray diffraction patterns for extruded corn masa samples prepared at different processing temperatures.

ition from an A to an A+V pattern, under the mechanic-thermal treatments in our extruder chamber, it is also clear (Fig. 3) that the intensity of the sharp peaks decreases with the increase in Tc. The latter indicates that the degree of crystallinity, that is the amount of starch in the semicrystalline phase, decreases with respect to that in the amorphous phase as indicated by the relative increase of the broad background. The crystallinity values obtained in the analysed samples are shown in Figure 6(b) and the value obtained for masa prepared by the traditional method was about 13%.

Figure 4 shows typical viscoamylographic profiles of maize powder samples before (raw corn) and after processed at 50, 65, 80 and 100 °C. These profiles show that changes in the starch slurry start to occur at a time of about 4·6 min, which correspond to temperatures in the range of 68–72 °C at which corn starch gelatinisation occurs. The viscosity first increases, indicating swelling and exudation of amylose of starch granules, then it reaches a maximum (peak viscosity), which depends on Tc, being higher for samples treated at lower Tc, due to a higher amount or percentage of starch granules available for gelatinisation.

Figure 5 shows the DDF as a function of frequency in the range of 1 to 25 Hz, for three selected masa samples processed at 60, 70 and 100 °C. As can be seen, in that frequency range the DDF has a peak whose maximum depends on Tc. The peak values for all measured masa samples are shown in Figure 6(d). A systematic study of the differential dissipation factor in samples with different characteristics, such as water content and Tc, indicated that this peak is related to changes in the polarisability of strongly bound water forming the first few layers onto the material[19] weakly bound water in the intergranular spaces and in the pores of the material has a resonance at much higher frequencies. The peak in the DDF assigned to strongly bound water, directly interacts with chemical groups in the molecules of the material. Thus, it is expected that the behaviour of so bound water is sensitive to structural changes and therefore to the gelatinisation of the starch in corn samples. Thermal diffusivity is basically a measure of the heating time within a given sample (the greater the thermal diffusivity the lower the heating time). In our experiment the thermal diffusivity [Figure 6(a)] has a maximum at around Tc = 80 °C. Thus, the increase in thermal diffusivity from Tc in the range of 50–80 °C indicates the formation of larger number of paths through which heat can be transmitted in the sample. The formation of a strong network in the material could be related to the cross-linking of starch granules by the exuded

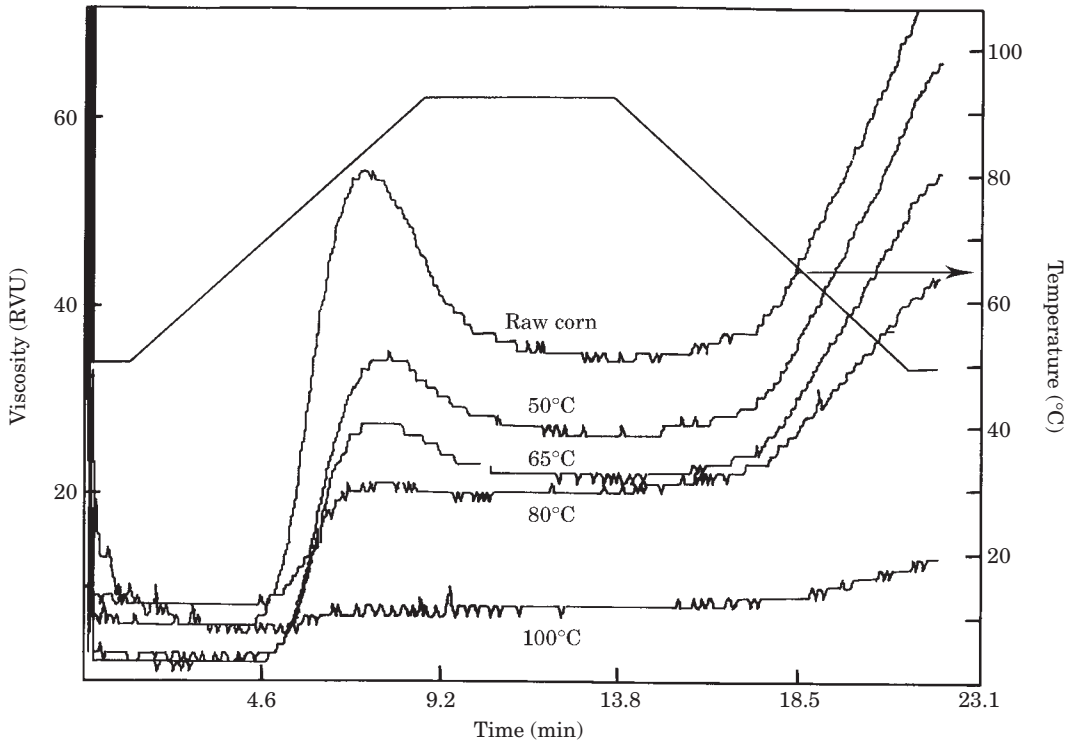

Figure 4 Typical viscoamylographic profiles of maize powder samples before (raw corn) and after processed at different processing temperature (1 RVU = 10cp).

Table I Textural characteristics of masa and tortilla produced of extruded corn masa processed at different temperatures

	Masa			Tortilla		
Temperature processing (°C)	Adhesiveness[a] (g_f)	Cohesiveness[b] (g_f)	Tensile strength[c] 1h (g_f)	Cutting force[d] 1h (kg_f)	Tensile strength[c] 24h (g_f)	Cutting force[d] 24h (kg_f)
70	22·5 c	111·0 a	943·7 a	7·871 a	1685·0 a	7·913 a
80	50·0 a	251·3 b	647·2 c	2·696 c	889·0 c	3·827 b
90	26·7 b	116·0 a	814·7 b	5·715 b	1444·0 b	7·610 a

Means with same letter, in same column, into each group are not significantly different ($P<0·05$).
[a,b]Tested with Texture Analyser, TA-XT2, using accessories TA-18.
[c,d]Tested with Texture Analyser, TA-XT2, using accessories TA-96 and TA-90, respectively.

amylose molecule during the heat treatment. The drop of thermal diffusivity with the increase in Tc above 80 °C is probably related to the dissolution of the granules occurring at these high temperatures. The X-ray data is shown in Figure 6(b) and the first point at the left side was taken from a sample of raw corn powder. As can be seen there is a slight increase in crystallinity when Tc is increased up to approximately 80 °C, followed by a pronounced drop for samples processed at higher Tc. The loss in crystallinity reflects the dissolution of the starch granules, which also produces the sudden drop in thermal diffusivity. For comparison, corn masa prepared using the traditional thermoalkaline method was also measured and the calculated crystallinity was approximately 13%.

Figure 6(c) shows the value of the peak viscosity in which the first point at the left side corresponds to the raw corn powder. This plot shows a continuous decrease in the peak viscosity up to a Tc of 80 °C, remaining approximately constant for higher Tc. Since the value of the peak viscosity is a measure of the starch granules that have been gelatinised and disrupted during the RVA

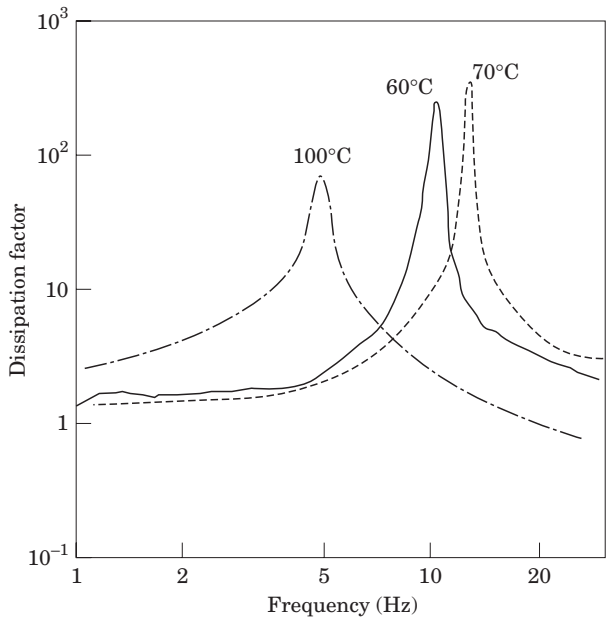

Figure 5 Differential dissipation factor as a function of extruded corn masa samples prepared at different processing temperature.

measurement, the decrease in peak viscosity indicates a complete gelatinisation and a continuous dissolution of starch granules during the extrusion process. At Tc above 80 °C most of the granules do not swell due to the damage by the high processing temperatures. For comparison the viscosity obtained for corn masa prepared used the traditional thermo-alkaline method was 14 RVU and the one for raw corn was 45 RVU. Similar to the thermal diffusivity, the position of the DDF peak as a function of Tc [Figure 6(d)] has its maximum value around 75 °C. Although the explanation of this behaviour is not quite clear at this point, it is significant that this maximum occurs at the temperature which separate the boundary between different behaviours in the other measured properties, showing the potential of this technique to monitor starch gelatinisation.

Table I shows some properties of masa and tortillas produced only with masa processed above 70 °C, because below that temperature it was not possible to form tortillas. Masa used to make tortillas has to develop the proper cohesiveness and adhesiveness without being sticky. In Table I is observed that the masa processed at 80 °C has the best consistency, that is minimum stickiness. Also, at this temperature the produced tortillas are smooth and the force required to cut them is the minimum. Similar behaviour is observed in

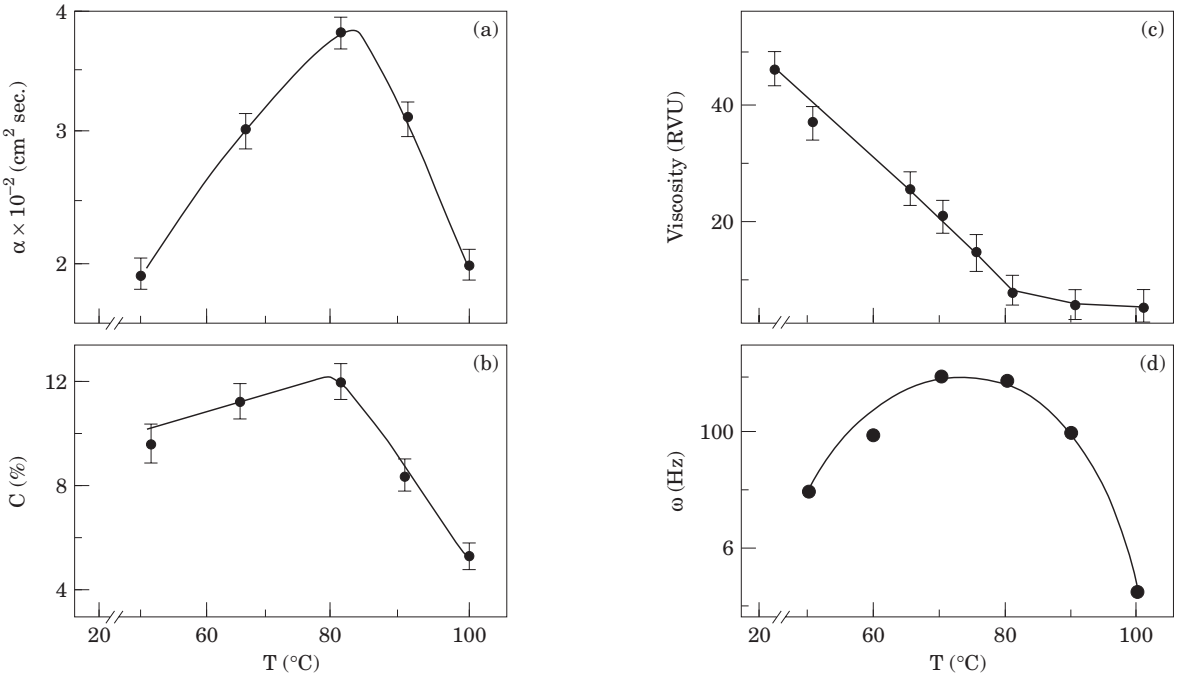

Figure 6 (a) Room temperature thermal diffusivity, (b) crystallinity, (c) peak viscosity and (d) frequency position of the maximum in the differential dissipation factor for corn masa samples prepared at different processing temperature.

tortillas measured immediately after they are made or in those tested 24h after were produced.

In summary, we have used the thermal, dielectric and rheological properties of corn masa prepared by extrusion at various processing temperatures to monitor the gelatinisation of corn starch and the textural characteristics of tortillas produced with these corn masas. The results show that there is a threshold temperature at about 80°C, above and below which the behaviour patterns of the measured properties are different. These properties were also measured in masa prepared by the traditional method (i.e. the thermo-alkaline method), and it is found that its values are very similar to those observed in extruded samples prepared at Tc of about 80°C. Considering that the optimum degree of starch gelatinisation for tortilla application is obtained in corn cooked using the traditional method, one can conclude that the degree of starch gelatinisation in the extruded masa is optimum at temperatures about 80°C. At 80°C the thermal diffusivity and the differential dissipation factor have maximum values. Textural characterisation made in tortillas produced with the extruded masa also indicate better mechanical properties for these obtained using masa processed at 80°C. This work also shows that the thermal diffusivity and DDF measurements are sensitive to the changes occurring during starch gelatinisation and could be used for this purpose.

Acknowledgements

We would like to thank E. Morales Sánchez for his technical assistance. One of the authors (G.V.A.) thanks the Consejo Nacional de Ciencia y Tecnología-México (CONACyT) for the financial support during the PhD studies.

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