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Experimental and modelling studies of the flow properties of maize and waxy maize starch pastes

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Abstract

Methods have been developed for characterising the time-dependent and non-Newtonian rheological behaviour of gelatinized maize starch pastes as a function of starch concentration, temperature and shearing conditions. Two types of commercial maize starch were used in this study: a normal maize with 30% amylose, and a waxy maize consisting mainly of amylopectin. Results from transient flow property measurements using a concentric cylinder viscometer show that the starch pastes are strongly thixotropic and shear thinning as characterised by a viscosity that decreases with time and rate of shear. The rheology of the starch pastes studied can be satisfactorily modelled using the structural kinetic approach which postulates that after gelatinization and cooling, the polysaccharide polymers and the swollen starch granules form network structures which are irreversibly broken under shear leading to the time- and shear-dependent change in the flow properties. At equilibrium, the starch pastes exhibit pseudoplastic behaviour which can be described by the power law model. While pastes of the two maize starches studied exhibit similar rheological behaviour at the same starch concentration and temperature, the normal maize starch paste is more viscous initially in the gel state, but breaks down more rapidly and becomes thinner than the waxy maize starch paste under the steady-state sheared condition. This difference is due to the different gel structures formed on cooling, and to the different swelling characteristics of the granules of the two maize starches studied.

Keywords: Maize starch paste; Waxy maize starch paste; Rheology of starch paste; Rheological characterization; Rheological modelling

1. Introduction

When a slurry of starch granules in water is heated beyond a critical temperature, the gelatinization temperature, the granules swell and the starch polysaccharides, amylose and amylopectin, are released and become solubilized. The resulting viscous paste is a dispersion of the swollen gelatinized starch granules in a macromolecular viscous solution. On cooling, if the paste is sufficiently concentrated, it can transform into a gel, consisting of the gelatinized starch granules embedded in an interconnected network of recrystallized polymer aggregates. Starch pastes and gels are therefore considered as composite materials whose rheological properties are determined by the contributions and interactions of both the dispersed phase (swollen starch granules) and the continuous viscous matrix [1,2].

The ability to form a viscous paste or gel with water by heating followed by cooling is the most important practical property of starch, and makes it suitable for numerous uses in both food and non-food industries. In many applications, the performance of starch products depends on their functional properties, which are determined largely by their rheological characteristics. A good knowledge of the rheology of the starch pastes and gels is thus essential for design of process equipment and for quality control and utilisation of starch products. The rheological behaviour of starch pastes and gels is usually complex and dependent on a wide range of factors such as botanical origin, starch concentration and pasting conditions (temperature, heating rate and agitation rate). In general, most starch pastes exhibit non-Newtonian flow behaviour with a viscosity which depends on time and rate of shear, shear history and temperature [1,3–6]. Some existing methods commonly used in the starch industry for measuring the viscous properties of starch pastes, however, are somewhat arbitrary and qualitative, such as the Scott test for hot paste consistency which yields the Scott viscosity

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number based on the time required to pour a known volume of the paste into a measuring cup [7]. The commercial starch viscometers, e.g., the Brabender amylograph and the Ottawa starch viscograph, are useful for studying the gelatinization and pasting behaviour of starch but the results obtained, in the form of viscograph patterns, are often system specific and can vary from one apparatus to another [3,8]. Recently, standard viscometric instruments using the rotational concentric cylinders and cone and plate geometries have been employed more frequently to characterise the rheological behaviour of starch pastes and gels of various origins [3–6]. The advantages of these instruments are that the shear fields are well defined and that the measurements are not single-point determination but can produce fundamental rheological properties in terms of shear stress–shear rate relationships which are independent of the type of the apparatus employed. Most previous investigations, however, have often used a measurement technique known as the 'hysteresis loop' method, in which the starch paste is subjected to successive cycles of increasing and decreasing shear rate to demonstrate the dependence of the flow properties on the rate and time of shear [3–6]. An example of the hysteresis of the rheological behaviour is shown in Fig. 1 for an aqueous waxy maize starch paste studied in this work. While these results are useful for demonstrating that the starch paste is non-Newtonian, shear-thinning and thixotropic, they can only be considered as qualitative because the hysteresis is strongly affected by the shearing cycle time, the past shear history and the maximum shear rate selected. As such, they cannot be used directly for engineering design and scale up of process and equipment.

The main objective of the present work is to develop a systematic and quantitative method for characterising the complex rheological behaviour of starch pastes and gels using both experimental and modelling approaches previously established for industrial slurries and suspensions. In this paper, the rheological behaviour of aqueous starch pastes prepared from two different industrial maize starches has been studied as a function of amylose/amylopectin content, starch concentration, temperature and shearing condition. From the results obtained, a rheological model for the starch pastes has been developed based on a consideration of the shear-induced change in the starch structure with time of shear.

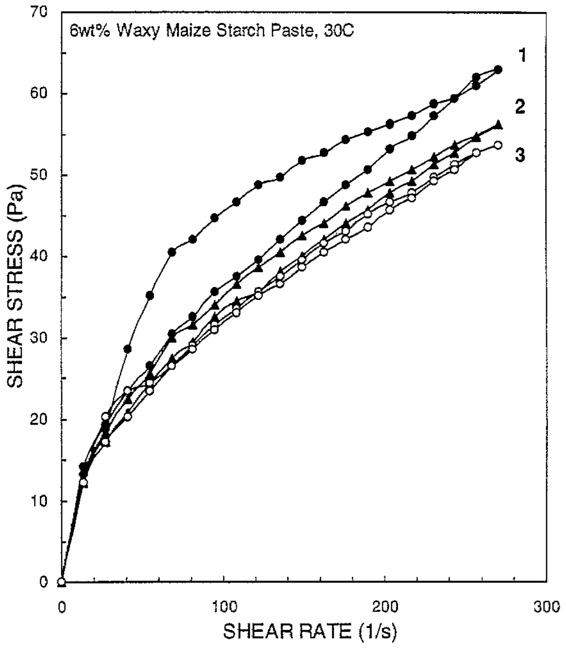

Fig. 1. Example of 'hysteresis' flow curves for a waxy maize starch paste tested in a concentric cylinder viscometer: (1) first, (2) second, (3) third shear cycle. Cycle time: 4 min.

2. Materials and methods

2.1. Materials and preparation

The starches used in this study were two types of food grade unmodified corn starch: a normal maize starch (brand name: Maize 3401C) and a waxy maize starch (Mazaca 3401X), both provided by Starch Australasia, New South Wales, Australia. The amylose to amylopectin proportion was about 30:70 for the regular maize and approximately 0:100 for the waxy maize. As received, the starches were in the form of granules with top sizes smaller than 15 μm, and moisture contents of 11–14%. Pasting of the starches was carried out by mixing a predetermined quantity of the starch granules in a volume of distilled water preheated to 50°C using a magnetic stirrer equipped with a hot plate. Once a homogeneous slurry had been obtained, the temperature was raised at a rate of 2°C/min to the gelatinization point, which was about 75°C for the waxy maize starch and 85°C for the regular maize starch. At this point, the waxy maize starch paste became a transparent viscous liquid which did not change significantly in appearance on cooling, whereas the regular maize starch paste exhibited a more milky colour and appeared less viscous, but formed an opaque smooth gel when cooled down. After gelatinization, the starch pastes were cooled down to room temperature (25°C) and rested for 2 h prior to flow property measurements. The effect of temperature on the rheological properties of the starch gels was studied by testing the starch samples reheated to 25, 35 and 50°C. All starch samples were prepared and used within 24 h then discarded and fresh pastes were prepared daily.

2.2. Flow property measurements

The basic rheological properties of the starch pastes were measured using a concentric cylinder viscometer (Haake Rotovisco, Model RV100) which has an inner cylinder rotating in a stationary outer cylinder. Two bobs with lengths of 60 mm and radii of 18.4 and 20.04 mm and a cup with a radius of 21.0 mm were used to form concentric cylindrical systems with radius ratios (cup-to-bob) of 1.05 and 1.14, respectively. The viscometer was equipped with a temperature vessel connected to a thermostatically controlled water bath to maintain the temperature of the test fluid constant during measurements. The experimental procedure employed with the viscometer for characterising the time-, shear-, and shear history-dependency of the rheological behaviour of the starch pastes consisted of a series of three measurements: (i) transient experiments at constant (steady) shear rates, (ii) experiments using step changes in shear rate and (iii) equilibrium flow property measurement at steady state.

The transient measurements in steady shear involved, first loading a fresh sample into the annular gap of the concentric cylinder viscometer. After a period of rest of 1 h, to allow the material to recover any damage to its structure incurred in the loading process, the sample was sheared at a constant shear rate, and the shear stress was measured as a function of time of shear until an equilibrium state was reached. The procedure was then repeated with other fresh samples at other constant shear rates. Typical time-dependent data are shown in Fig. 2 as plots of the apparent shear viscosity, defined as the ratio of shear stress to shear rate, vs. time of shear for different constant shear rates.

In the step-change-in-shear-rate method, the starch paste was first sheared at a fixed shear rate as in the steady shear experiment. As soon as steady state, or equilibrium, was established, the shear rate was either increased or decreased to another level and the shear stress was recorded as a function of time. After equilibrium was attained at the new shear rate, the latter was returned to the initial level and the above procedure was repeated with another step change (increase or decrease) in shear rate. This type of experiment is useful for studying the effect of previous shear history on the flow behaviour of time-dependent fluids [9].

Measurements of the steady-state or equilibrium flow properties were made on starch paste samples which had been subjected to a long preshearing period to ensure that the materials reached an equilibrium structured and viscous state at which the rheological behaviour is no longer dependent on time of shear. Based on the steady-shear and step-change-in-shear-rate measurements, we found that preshearing the starch paste at a shear rate of 255 s^{-1} in the viscometer for 2 h was sufficient to break down the material structure to equilibrium and remove the thixotropic effects. In addition to the above measurements by means of the concentric cylinder viscometer, the vane–shear method was also employed with the presheared starch samples to detect and measure the yield stress property, which is a characteristic of viscoplastic fluids [10,11].

3. Results

3.1. Time-dependent flow properties

Both the regular and waxy maize starch pastes studied, after cooled down from their gelatinization temperatures, exhibit the characteristics of highly time-dependent (thixotropic) materials. Typical experimental results obtained from the time-dependent steady shear experiments are shown in Figs. 2–4 in terms of the apparent viscosity, defined as the ratio of shear stress to shear rate, plotted as a function of time of shear at constant shear rates. At a fixed shear rate, the apparent viscosity decreases rapidly with time within about the first 10 min of shear and approaches a constant value corresponding to an equilibrium state after approximately 1 h. The rate and extent of viscosity reduction appear to depend on the type of starch, starch concentration, the applied shear rate and temperature. The marked difference in the time-dependent flow properties of the two maize starch gels may be seen in Fig. 2. At the same starch concentration (6 wt.%) and temperature (25°C), the regular maize starch paste is considerably more viscous than the waxy maize paste in the presheared, undisturbed state. However, in the presence of shear the regular maize starch paste 'breaks down' more rapidly with time towards an equilibrium or steady-state viscosity which is lower than that of the waxy maize starch paste.

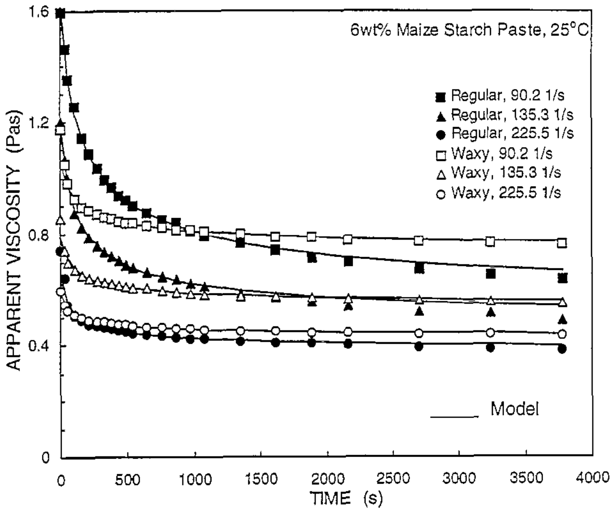

Fig. 2. Transient viscosity data at constant shear rates for 6 wt.% maize starch pastes at 25°C. Effect of starch type.

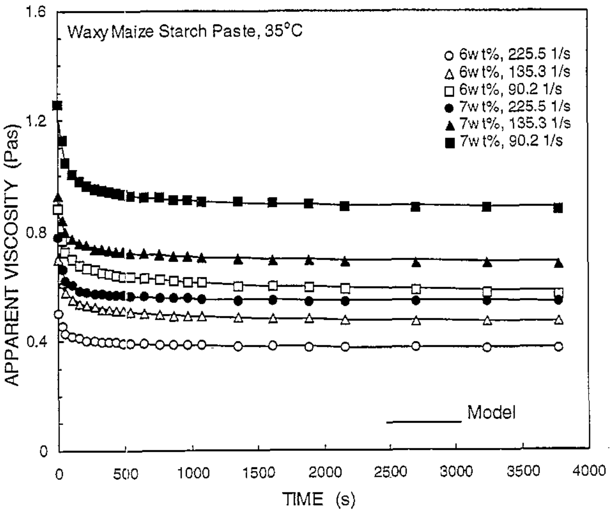

Fig. 3. Transient viscosity data at constant shear rates for waxy maize starch pastes at 35°C. Effect of starch concentration.

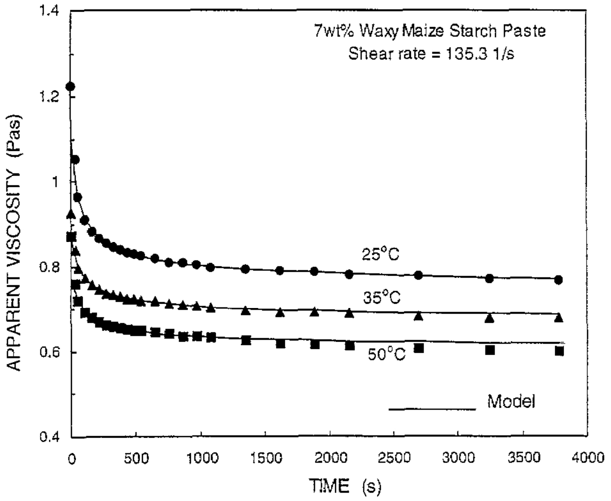

Fig. 4. Transient viscosity data at a constant shear rate ($135\ \mathrm{s}^{-1}$) for 7 wt.% waxy maize starch pastes. Effect of temperature.

The difference in the time-dependent flow behaviour of the two maize starches studied was observed at all starch concentrations and temperatures investigated. The effect of starch concentration is shown in Fig. 3 for the waxy maize starch paste at two concentrations, 6 wt.% and 7 wt.%, and at a temperature of 35°C. Even within this small range of concentration, it is found that an increase in the starch content obviously leads to an increase in the paste viscosity but changing starch concentration does not appear to significantly affect the thixotropic characteristics of the paste. Fig. 4 demonstrates that temperature has a dominant effect on the viscosity and the thixotropic behaviour of the maize starch pastes.

Experimental results from the step-change-in-shear-rate experiment indicated that after the starch paste had been sheared to equilibrium at a given shear rate, it did not regain its original structure when the applied shear rate was reduced from the initial level. This may imply that the structural breakdown process taking place in the starch paste during shear is irreversible and that the rebuilding of the thixotropic structure of the sheared paste during shear is slow or negligible. Similar observations with a corn starch paste have been reported by Ref. [5].

3.2. Modelling of time-dependent flow behaviour

The observed time-dependent flow behaviour of the starch pastes studied may be modelled using the structural kinetic approach, which has been successfully employed for minerals and other industrial suspensions [9,12]. This model postulates that the change in the rheological properties is associated with shear-induced breakdown of the internal fluid structure in the starch paste. Using the analogy with chemical reactions, we may express the structural breakdown process as

$$(\text{Structured}) \rightarrow (\text{Non-Structured})$$

The rate of breakdown of the structure during shear depends on the kinetics of the above 'reaction'. Based on the experimental results from the transient measurements at constant shear rates, viz. Figs. 2–4, and from the step change in shear rate measurements, it may be assumed that the thixotropic structure in the starch pastes breaks down irreversibly without significant buildup. Let $\psi = \psi(\dot{\gamma}, t)$ be a dimensionless parameter representing the structured state at any time t and under an applied shear rate $\dot{\gamma}$, the rate of structural breakdown may be expressed as

$$-\mathrm{d}\psi/\mathrm{d}t = k(\psi - \psi_\infty)^m \tag{1}$$

where $k = k(\dot{\gamma})$ is the rate constant, a function of shear rate ($\dot{\gamma}$), and m is the order of the breakdown 'reaction'. Initially, at the fully structured state, $t = 0$: $\psi = \psi_0$, and at steady state, $t \rightarrow \infty$: $\psi = \psi_\infty$.

At a constant applied shear rate, integration of Eq. (1) from $t = 0$ to t yields

$$(\psi - \psi_\infty)^{1-m} = (m-1)k \cdot t + (\psi_0 - \psi_\infty)^{1-m} \tag{2}$$

To apply Eq. (2) to the experimental transient viscosity data, a relationship between ψ and measurable rheological quantities needs to be specified. We may define ψ in terms of the apparent viscosity ($\eta = \tau/\dot{\gamma}$) as

$$\psi(\dot{\gamma}, t) = (\eta - \eta_\infty)/(\eta_0 - \eta_\infty) \tag{3}$$

where η_0 is the initial apparent viscosity at $t = 0$ ('structured' state), and η_∞ is the final or equilibrium apparent viscosity at $t \rightarrow \infty$ (equilibrium structured state). Both η_0 and η_∞ are functions of the applied shear rate only.

Substituting Eq. (3) into Eq. (2) we obtain, for a fixed shear rate:

$$(\eta - \eta_\infty)^{1-m} = [(m-1)k \cdot t + 1](\eta_0 - \eta_\infty)^{1-m} \tag{4}$$

The form of Eq. (4) allows a simple way for testing the validity of the model and determining of the model parameters m and k. Note that Eq. (4) is valid only under the constant shear rate condition.

For all the starch pastes studied, we found that their transient viscosity data at constant shear rates could be satisfactorily correlated using $m = 3$, i.e., with a third-order irreversible kinetic model. This is illustrated in Fig. 5 for a waxy maize paste where plots of $(\eta - \eta_\infty)^{-2}$ vs. t are linear, which confirm the applicability of the model according to Eq. (4). An excellent comparison between the model-fitted results (continuous lines) and the experimental viscosity–time data may be seen in Figs. 2–4. Table 1 summarises values of the rate constant, k, which is an indication of the rate of thixotropic breakdown, and the ratio of the initial to equilibrium viscosity, η_0:η_∞, which may be considered as a relative measure of the extent of thixotropy, as a function of starch type, starch concentration, temperature and shear rate. It may be seen that for a given starch, k generally increases with increasing shear rate, temperature and concentration, as we would expect for thixotropic structured fluids. Under the same condition, the regular maize starch paste has a higher k value and a greater η_0:η_∞ than those of the waxy starch,

Table 1
Structural breakdown rate constant (k) and viscosity ratio (η_0/η_∞) for the third-order irreversible kinetic model

Starch	Paste	wt.%, Starch	T (°C)	$\dot{\gamma}=225.5\ s^{-1}$		$\dot{\gamma}=135.3\ s^{-1}$		$\dot{\gamma}=90.2\ s^{-1}$	
				$k\ (s^{-1})$	η_0/η_∞	$k\ (s^{-1})$	η_0/η_∞	$k\ (s^{-1})$	η_0/η_∞
Regular	maize	6	25	0.015	1.79	0.008	2.66	0.006	3.17
Regular	maize	6	35	0.015	1.52	0.013	2.13	0.011	1.73
Waxy	maize	6	25	0.008	1.24	0.006	1.49	0.004	1.39
Waxy	maize	6	35	0.011	1.41	0.009	1.40	0.008	1.45
Waxy	maize	7	25	0.016	1.39	0.014	1.49	0.014	1.46
Waxy	maize	7	35	0.037	1.39	0.012	1.30	0.021	1.42
Waxy	maize	7	50	0.044	1.20	0.011	1.29	0.006	1.35

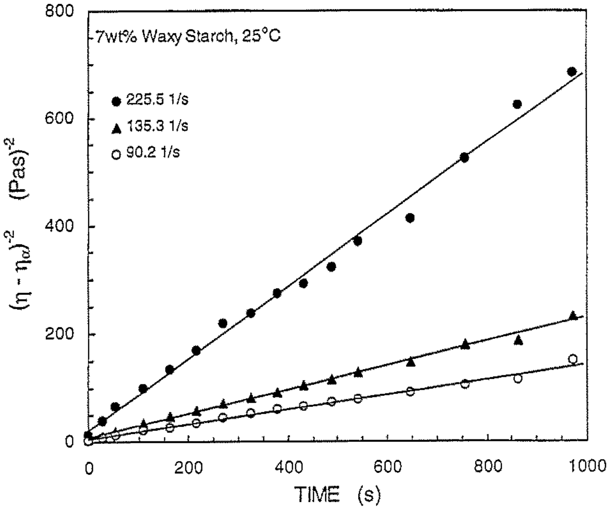

Fig. 5. Testing of the structural kinetic model, Eq. (4), with 7 wt.% waxy maize starch pastes at 25°C.

indicating that the former exhibits a more shear-sensitive thixotropic nature.

3.3. Steady-state flow properties

For the equilibrium (steady-state) flow property measurement, the starch paste samples were presheared at a maximum shear rate of 255 s^{-1} for 2 h in order to break down completely the thixotropic structure. Subsequent measurements at this and lower shear rates gave us the information on the equilibrium behaviour of the fluid with minimum time-dependent effects. The flow properties for all the starch pastes measured at the steady-state condition are shown in Figs. 6 and 7 as plots of shear stress vs. shear rate. The flow curves (on linear scales) demonstrate that the pastes are shear-thinning with the presence of a possible yield stress, which is the shear stress intercept at zero shear rate. A direct measurement of the yield stress using the vane–shear method, however, did not produce finite yield values for the starch pastes previously sheared to the steady equilibrium state. Thus, based on this result and the equilibrium flow property data in Figs. 6 and 7, it may be concluded that the starch pastes studied under the steady-state sheared condition may be considered as pseudoplastic fluids without a measurable yield stress. Furthermore, the measured flow behaviour over the range of shear rates employed may be described by the power law model:

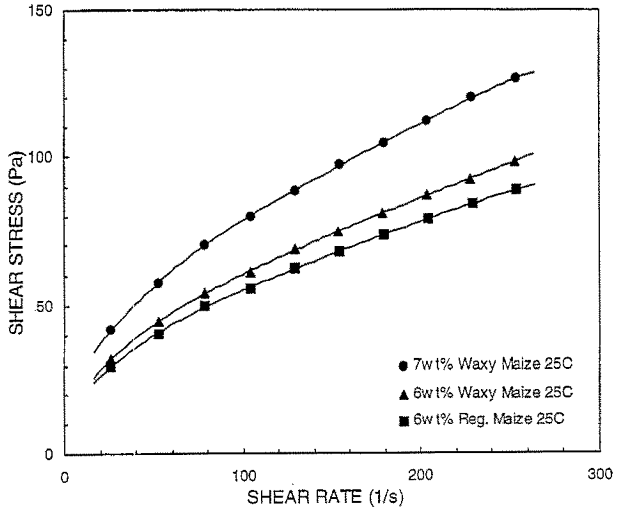

Fig. 6. Equilibrium flow properties of 6 wt.% and 7 wt.% maize starch pastes at 25°C.

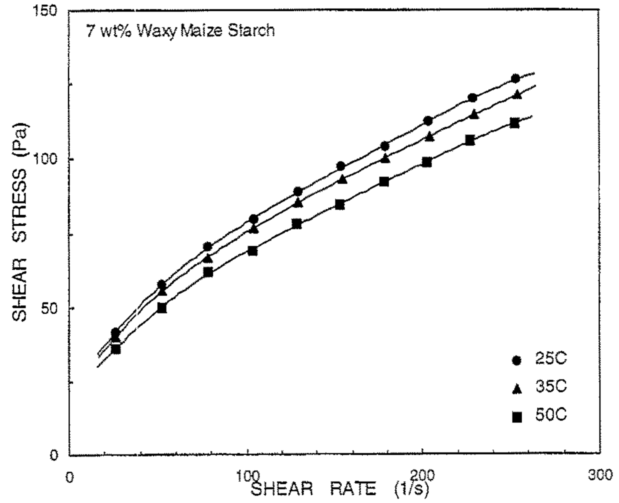

Fig. 7. Equilibrium flow properties of 7 wt.% waxy maize starch pastes. Effect of temperature.

$$\tau = K\dot{\gamma}^n \qquad (5)$$

Table 2
Values of power-law constants (K) and (n)

Starch		(wt.%) Starch	T (°C)	K (Pa s^{1+n})	n (−)
Regular	maize	6	25	6.45	0.49
Regular	maize	6	35	5.60	0.48
Waxy	maize	6	25	7.18	0.42
Waxy	maize	6	35	6.12	0.48
Waxy	maize	7	25	8.49	0.48
Waxy	maize	7	35	8.26	0.48
Waxy	maize	7	50	7.19	0.49

Values of the two model constants, K and n, are summarised in Table 2. It is found that the value of K, a measure of viscosity, is very dependent on type of starch, concentration and temperature. On the other hand, n, a measure of the non Newtonian behaviour, is essentially constant (~0.48) and independent of the three factors. Ref. [13] reported similar results ($n = 0.4$–0.6) with gelatinized maize and wheat starch pastes.

4. Discussion

The results from our measurements of the shear rheological properties and modelling study confirm that the maize starch pastes studied exhibit non-Newtonian flow behaviour, as characterised by a viscosity that decreases with time and rate of shear (shear-thinning or pseudoplastic). These are typical properties of composite two-phase fluids with internal structures which undergo shear induced changes with time, leading to changes in the macroscopic flow behaviour. A good knowledge of the nature of the structures present in the two maize starch pastes studied is thus useful for the understanding and interpretation of their rheological characteristics. The regular maize starch granules contain some 30% of their polysaccharides as amylose, with the remainder as amylopectin. Amylose is essentially a high molecular weight, linear-chain polymer, which may be readily leached out and solubilized in water during the gelatinization process, forming a viscous continuous phase in which the swollen granules, consisting mainly of amylopectin, are suspended. On cooling, the amylose in solution undergoes retrogradation in which its molecules align themselves and form association by mutual side bonding, leading to formation of insoluble aggregates [13–15]. At sufficiently high concentrations, these aggregates extend throughout the whole volume, entrapping the aqueous medium and the swollen starch granules, and forming a gel. In contrast to the regular maize starch, the granules of the waxy maize starch contains only amylopectin, which is known to be less susceptible to retrogradation and does not form gel on cooling due to the highly branched structure of the amylopectin polymers [16]. The observed gel-like behaviour of the gelatinized waxy maize starch paste is possibly caused by the considerable swelling ability of the waxy starch granules as compared to the common cereal starches [15]. The large swollen gelatinized starch granules may be so close together that network structure formation is facilitated by association of the short outer branches of the amylopectin molecules [3,15]. While the gel micro-structures in the regular and waxy maize starch pastes are formed by different mechanisms, our experimental and modelling results (Fig. 5) indicate that they both break down irreversibly under shear following a third-order structural kinetics. This suggests that both types of structure break down by rupture of the same kind of network bonds, which may simply be hydrogen bonding of the hydroxyl groups on adjacent amylose chains in the regular maize, and on the neighbouring outer short chains of amylopectin in the waxy maize. The rate of structural breakdown, however, is faster in the regular maize than in the waxy maize, as seen from the time-dependent viscosity data in Figs. 2–4 and from the values of the breakdown rate constant k in Table 1. This may indicate that the network bonds found in the outer amylopectin chains in the waxy maize starch paste are fewer and more rigid than those existing along the linear amylose chains in the regular maize gel.

With prolonged shearing, the gel network is broken down to an equilibrium state, at which the starch paste may be considered as a suspension of swollen starch granules in a viscous solution. As the typical results in Figs. 6 and 7 show, both maize starch pastes exhibit the pseudoplastic, or shear-thinning, flow behaviour with an apparent viscosity that decreases with increasing shear rate. This type of rheological behaviour is typical of liquid emulsions and is expected to depend on the viscosity of the solution and the concentration, effective hydrodynamic volume, and rigidity of the swollen granules. Thus at a fixed starch concentration, the waxy maize starch paste at equilibrium is more viscous than the regular maize starch paste because the swollen granules in the former are larger and occupy a greater volume than those granules dispersed in the regular maize starch paste (see Figs. 2 and 6).

The structural kinetic approach for modelling thixotropy employed here seems to describe satisfactorily the experimental time-dependent flow property data at constant shear rates for all the maize starch pastes studied (see Figs. 2–4). In the present formulation, the dimensionless structure parameter (ψ), which represents the state of the thixotropic structure, is defined based on the apparent shear viscosity, via Eq. (3), simply because the viscosity can be directly obtained from the transient experiments and that it includes the effects of both the shear rate and time of shear. The use of other rheological properties for the definition of the structure parameter would require some rheological model be assumed for the starch pastes. While the modelling approach employed in this work is a phenomenological one, we believe it is a good starting point for understanding and interpreting the time-dependent changes in the measurable rheological properties in terms of the shear-induced change of some internal structure in the fluid. The knowledge gained will form a basis for the development of a more complex model in which the nature of the microstructure of the fluid is taken into account.

5. Conclusions

Using the experimental methods developed, the rheological behaviour of aqueous starch pastes of two types of maize starch can be characterised as strongly thixotropic and shear thinning (pseudoplastic). The time-dependent rheology of the starches studied has been satisfactorily modelled by postulating that the internal gel structure formed by the polysaccharide polymers and the swollen starch granules breaks down irreversibly under shear following a third-order kinetics. At equilibrium, the starch pastes exhibit pseudoplastic behaviour which can be described by the power law. The type of starch, viz. the amylose/amylopectin proportion, starch concentration and temperature all have effects on the rheological behaviour of the starch pastes studied.

Acknowledgements

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