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A rheological characterization of semi-solid dairy systems

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Abstract

Dispersions of cross-linked starch in full fat milk, taken as models of custard model systems, have been characterized by different rheological means: viscoelastic measurements, classical flow measurements and 'vane' rheometry. From viscosity measurements, the flow behaviour was described within the shear rate range 0.01–100 s^{-1}. The flow curves were fitted using the Herschell–Bulkley equation over the shear rate range 0.1–100 s^{-1} while a deviation was found towards the low shear rate range, making the determination of the yield stress non realistic. Instead, measurements with the 'vane' device in low shear conditions provided a way to estimate the yield stress, at rest and after shearing, but the entire flow curve was not described. From the viscoelastic measurements at low strain amplitude, the mechanical spectra were obtained. Linearity tests beyond the linearity limits provided the critical stress corresponding to the G'–G'' cross-over. The parameters obtained from these different rheological methods are discussed.

Keywords: Cross-linked starch; Rheology; Viscometry; Custard; Viscoelasticity; Vane rheometry

1. Introduction

Many commercial dairy desserts consist classically of milk mixed with starch to which a gelling agent such as carrageenan is added. These systems can have different designations depending on the country, such as 'vla', 'natillas', 'crème-dessert' or 'custard sauce'. A model system derived from such a typical formula has been suggested within the framework of the COST 921 Action. This was chosen since relatively easy to prepare in a reproducible way, easily consumed and its composition was close to that of real foods. It was agreed that a complete rheological characterization should be performed in order to be able to make comparisons between the different teams of the program.

From the basic composition of these systems, it is clear that starch plays a major role in the properties of the system. However, the other components such as milk proteins, fat, other polysaccharides are involved in the final texture of the dispersion. Basically, these dairy systems are composed of full fat milk to which a cross-linked starch (from tapioca in the present work) is added together with a very low amount of a gelling carrageenan and in the presence of sucrose. As a result, a custard can be merely described as a suspension of deformable particles (the swollen starch granules) dispersed in a continuous medium containing milk proteins as well as the carrageenan (van de Velde, van Riel, & Tromp, 2002; van de Velde, Weinbreck, Edelman, van der Linden, & Tromp, 2003).

Recently, several studies dealing with the rheological characterization of custard have been performed (de Wijk, Prinz, & Janssen, 2006; de Wijk, Terpstra, Janssen, & Prinz, 2006; González-Tomás, Bayari, Taylor, & Costell, 2007; Jellema, Janssen, Terpstra, de Wijk, & Smilde, 2006; Tárrega, Vélez-Ruiz, & Costell, 2004; Tárrega, Durán, & Costell, 2005; Tárrega, Durán, & Costell, 2004; Veléz-Ruiz, González-Tomás, & Costell, 2005). In most of these studies, the aim was to establish the link between texture attributes from sensory analysis and rheological data. The rheological parameters that were considered were obtained either from flow measurements or from viscoelastic determinations. Viscosity measurements were also performed with the 'vane' geometry in order to estimate the yield stress. This technique has been claimed to be particularly useful in the case of

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suspensions and is becoming popular for that purpose (Barnes & Nguyen, 2001). It has been applied to starch suspensions (Genovese & Rao, 2003) and also to custard (Tárrega, Costell, & Rao, 2006).

In the present study, the rheological properties of the model systems have been assessed by three different methods. First, the viscoelastic properties have been described in oscillatory shear from the hot state (60 °C) to the cooled state (15 °C). This allowed one to estimate the properties in unperturbed conditions. Second, classical flow measurements (viscometry) were performed at 15 °C. By this way, the 'shear sensitivity' ('thixotropic') properties as well as the shear-thinning behaviour could be estimated. It was also possible to determine the yield stress, that is a parameter useful to characterize the behaviour at a low shear rate. Lastly, viscosity measurements were carried out using the 'vane' geometry. The information taken from these different ways will then be discussed.

2. Experimental

2.1. Materials

A cross-linked tapioca starch (Farinex VA 60 T) from AVEBE (NL) has been used. The κ-carrageenan sample (Meypro$^{\text{TM}}$ lact HMF) was obtained from Gelymar (CH). A commercial UHT sterilized full fat milk was used for all the preparations.

2.2. Methods

2.2.1. Preparation of the dispersions

The dispersions were prepared following the procedure agreed within the COST 921 programme. κ-carrageenan, starch and sucrose (5%) were dispersed in full fat milk. The mixture was then placed in a water bath at 98 °C for 50 min and stirred at 300 rpm. Four different preparations were compared by varying the starch content (3.5% or 4%) and the κ-carrageenan content (0% or 0.01%). After the samples have been prepared, they were placed in the cup of the measuring system of the rheometer at 60 °C.

2.2.2. Flow measurements and viscoelastic measurements

These were performed with a controlled stress rheometer (AR 2000 from TA Instruments) using a coaxial cylinders device (internal diameter 14 mm; external diameter: 15 mm; height: 42 mm). The sample was placed hot (temperature > 60 °C) in the measuring device immediately after the preparation.

A first mechanical spectrum (G' and G'' as a function of angular frequency) was obtained at 60 °C. Then, a temperature cycle (1 °C/min) from 60 °C down to 15 °C, then from 15 °C up to 60 °C and back to 15 °C was performed in order to evaluate the temperature dependence. This was followed by measurements of G' and G'' as a function of frequency (mechanical spectrum) at 15 °C. At the end, a linearity test (with shear strain varying from 0.02% to 500%) again at 15 °C, was performed. For all the measurements, the linearity limit was of the order of 1%. In all experiments, the measurements were performed at a 1% shear strain, unless specified.

At the end of these viscoelastic measurements, flow measurements were performed at 15 °C. First, the 'thixotropic' behaviour was described by programming a shear rate cycle between 0 and 100 s^{-1} for 2 × 2 minutes. This allowed one to estimate the shear sensitivity of the suspension. Then steady shear measurements were carried out by a stepwise programmation of the shear rate from 100 s^{-1} to 0.01 s^{-1}. This allowed one to describe the flow behaviour after the sample has been sheared at high shear rate (100 s^{-1}). Lastly, a new linearity test was performed and the viscoelastic behaviour of the sheared suspension at 15 °C was estimated. All measurements were performed in duplicate. Reproducibility between measurements was of the order of 5%.

2.3. Vane rheometry

The measurements were performed using the AR 2000 rheometer equipped with a 6-blade vane impeller (vane diameter: 12.5 mm; cup diameter: 15 mm; height: 33 mm). The procedure was similar to that used by Tárrega et al. (2006): the torque was measured as a function of time with the vane rotating at 0.05 rpm. The shear stress was calculated from the classical equation employed for the vane geometry (Barnes & Nguyen, 2001; Genovese & Rao, 2003):

$$\tau = \frac{T}{K}$$

with

$$K = \frac{\pi D^3}{2}\left(\frac{H}{D} + \frac{1}{3}\right)$$

where T is the torque, τ is the shear stress, D and H are the vane and height diameter, respectively. The shear rate ($\dot{\gamma}$) is obtained by the following equation:

$$\dot{\gamma} = k_s \frac{N}{60}$$

with N the rotation speed and where k_s is a constant which has been determined using a fluid of known viscosity.

The sample was then sheared at high speed (260 rpm) before a new test at 0.05 rpm was performed. This procedure allowed one to get information at low shear (0.05 rpm corresponding to a shear rate of 0.021 s^{-1}) on the undisturbed sample (before shearing) and then on the destructured system (after shearing).

3. Results and discussion

3.1. Viscoelastic behaviour

The viscoelastic properties at 15 °C of the different compositions are illustrated in Fig. 1. The variations of the storage modulus (G') and of the loss modulus (G'') as a function of angular frequency are plotted in the frequency range 10^{-1}–100 rad/s. In Fig. 1a, the two suspensions with 3.5% starch are compared. When starch was only present, the G' and G'' moduli slightly increased with frequency, G' was higher than G'', and the difference tended to increase as the frequency decreased. Moreover, G' tended to level of as the frequency decreased. Such a frequency dependency of G' and G'' is typical of a solid-like behaviour and has been reported in the case of starch suspensions when the volume fraction occupied by the swollen starch granules is higher than 50–55% (Tecante & Doublier, 1999). The present results obtained for a suspension of a cross-linked tapioca starch in full fat milk are similar to those reported for a suspension of a cross-linked waxy starch in water, the only difference likely arising mainly from a difference in the cross-linking degree of the starch samples. Such a solid-like behaviour is observed when the packing of the swollen starch granules is high enough to occupy a large part of the volume (typically more than 50–55%). The effect of the presence of a low amount of κ-carrageenan (0.01%) is also illustrated in Fig. 1a. The moduli were dramatically shifted to higher values while the overall mechanical spectrum remained quite comparable : G' at 1 rad/s was increased from 12 Pa in the case of starch alone to 58 Pa when 0.01% of κ-carrageenan have been added (Table 1). Meanwhile, the elastic character was reinforced, as illustrated by the G''/G' ratio which decreased from 0.53 in the case of starch alone down to 0.25 for starch + κ-carrageenan. In Fig. 1b, the mechanical spectra for 4% starch concentrations with or without 0.01% κ-carrageenan, respectively, are showed. The viscoelastic behaviour was accentuated but displayed similarities with respect to 3.5% starch concentration. Again, when κ-carrageenan was present the elastic character of the suspension was reinforced:G' at 1 rad/s increased from 56 Pa ($G''/G' = 0.28$) in the absence of κ-carrageenan up to 185 ($G''/G' = 0.15$) in the presence of this polysaccharide. From these simple observations, the importance of the κ-carrageenan on the rheological properties, and hence on the texture, of a custard-type model system is clearly evidenced.

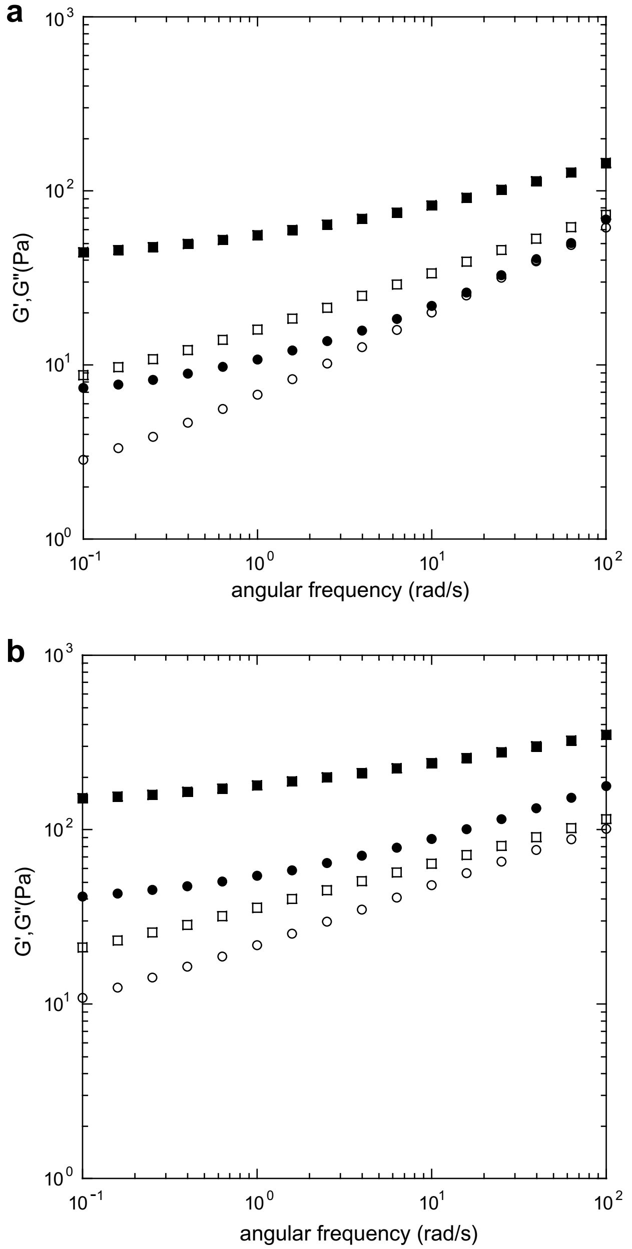

Fig. 1. G' and G'' as a function of angular frequency: (a) 3.5% starch (G'(●), G''(○)); 3.5% starch + 0.01% κ-carrageenan (G'(■), G''(□)); measurement temperature: 15 °C and (b) 4% starch (G'(●), G''(○)); 4% starch + 0.01% κ-carrageenan (G'(■), G''(□)); measurement temperature: 15 °C.

In Fig. 2, the temperature dependence ('down' and 'up' curves between 15 °C and 60 °C) of the 4% starch concentrated systems is illustrated. In the case of starch alone, the difference between 60 °C ($G' = 12$ Pa) and 15 °C ($G' = 50$ Pa) was limited; on another hand, the 'down' and the 'up' curves were superimposed. This evidences the complete thermoreversibility of the system. Thermoreversibility was also found when κ-carrageenan was present; however, the 'down' curve was below the 'up' curve indicating thermal hysteresis. Moreover, the difference between 60 °C ($G' = 30$ Pa) and 15 °C ($G' = 165$ Pa) was greater. This likely reflects the presence of a carrageenan network despite the very low concentration of this polysaccharide. Similar results could be observed with 3.5% starch.

The linearity tests have been carried out in order to determine the limits of the linear behaviour. Measurements were performed at 1 rad/s. Two examples are given in Fig. 3 for the measurements at 15 °C in the case of 4% starch with or without κ-carrageenan. These curves show the variations of G' and G'' as a function of stress. Deviation from linearity took place for a stress of about 1.73 Pa in the presence of κ-carrageenan and 0.54 in the case of starch alone. In both cases, this corresponded to a shear strain of about 1%. This means that the viscoelastic measurements have to be performed at a shear strain below 1%. In these curves, we also can determine the G'–G'' crossover in terms of stress amplitude. In this example, this was

Table 1
Main parameters obtained using the different rheological methods

Measurements		3.5% starch	3.5% starch + 0.01% κ carrag.	4% starch	4% starch + 0.01% κ carrag.
Viscoelastic measurements	G',G'' (Pa) at rest[a,b]	11.9, 0.25	58.4, 14.7	57.0, 15.8	185, 33.3
	Shear stress at the G'-G'' cross-over at rest (Pa)[a]	0.94	8.33	4.90	32.0
	G',G'' (Pa) after shearing[a,b]	21.5, 6.3	41.9,14.6	59.9, 22.7	129, 30
	Shear stress at the G'-G'' cross-over after shearing (Pa)[a]	1.28	2.80	3.02	7.9
Flow curve (fit with the Herschell–Bulkley equation)	τ_0 (Pa)	1.36	2.36	2.91	6.36
	'n'	0.524	0.518	0.510	0.501
	K_1 (Pa)	3.43	5.06	7.10	12.0
Flow curve (fit with the Casson equation)	τ_0 (Pa)	0.48	1.0	1.0	2.25
	η_P (s^{-1})	7.56	10.4	17.8	37.7
'Vane' Rheometry	τ_{0S} (Pa)	1.45	11.6	7.65	29.4
	τ_{0D} (Pa)	2.91	4.16	6.61	8.40

[a] Measurements at 1 rad/s.
[b] In the linearity limits ($\gamma_0 \leqslant 1\%$).

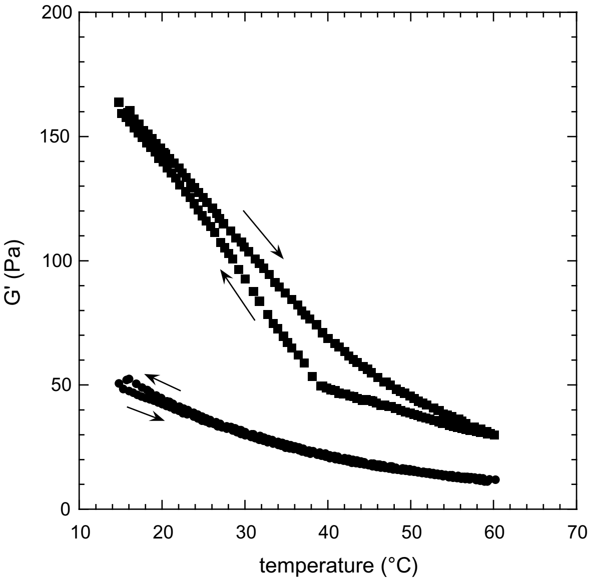

Fig. 2. G' as a function of temperature: 4% starch (G'(●)); 4% starch + 0.01% κ-carrageenan (G'(■)); frequency: 1 rad/s.

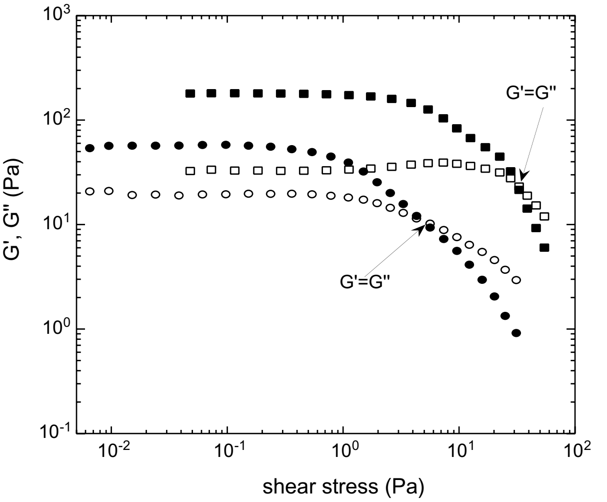

Fig. 3. Linearity experiment. G' and G'' as a function of shear stress: 4% starch + 0.01% κ-carrageenan; G'(●), G''(○) before shearing; G'(■), G''(□) after shearing; measurement temperature: 15 °C.

of 4.9 Pa in the case of starch alone and 31 Pa in the presence of κ-carrageenan. The overall results are given in Table 1.

3.2. Flow behaviour

In Fig. 4, the flow curves (shear stress vs shear rate) of the different suspensions, 3.5% (Fig. 4a) and 4% (Fig. 4b), respectively, are showed. These curves have been obtained by linearly programming the shear rate from 0 to 100 s^{-1} and then back to 0, the entire measurement lasting 2×2 min. They are plotted in linear scales and allow one to estimate the possible shear sensitivity of the system. With starch alone, the 'up' curve and the 'down' curves were superimposed. These curves illustrate the non-newtonian and shear-thinning behaviour classically exhibited by concentrated starch suspensions. The fact that no hysteresis is found means that shearing at high shear rate does not modify the structure of the system. When dealing with the complete formula, κ-carrageenan being incorporated, the shear-thinning behaviour was still observed. In addition, an hysteresis loop was seen, the 'up' curve being above the 'down' curve. This hysteresis indicates that shearing at high shear rate modifies the structure of the system, by decreasing its viscosity. Again, the present results are likely to be ascribed to the presence of the κ-carrageenan network in the system. From these viscosity measurements, it is also clear that a significant increase of the viscosity is brought about by the presence of the low amount of κ-carrageenan.

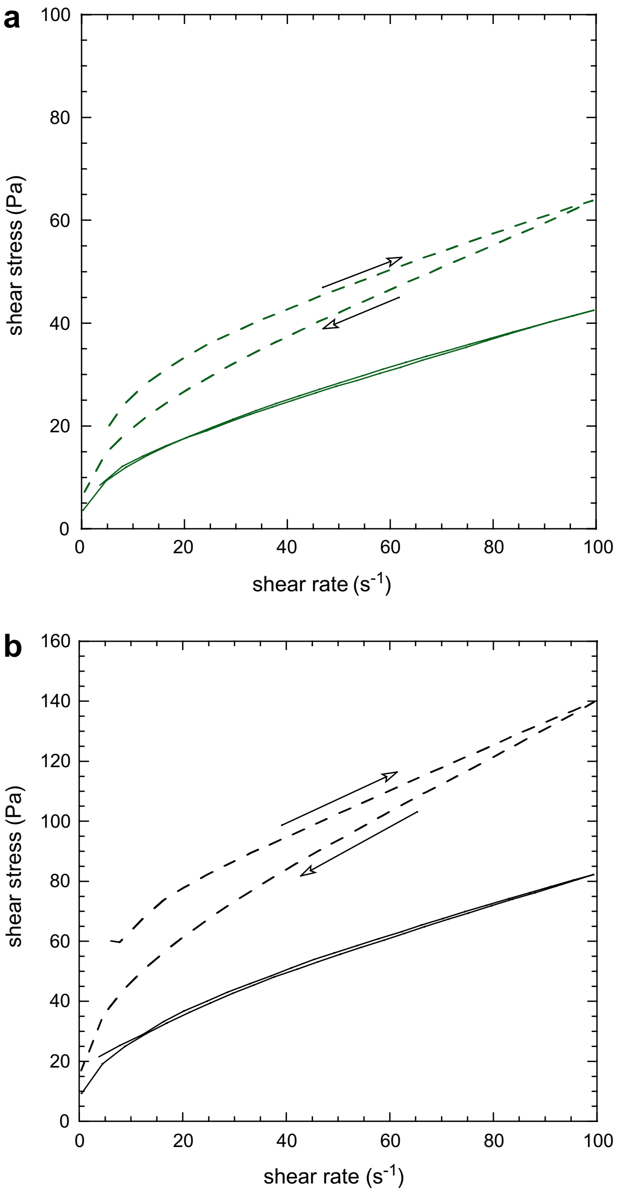

Fig. 4. Flow curves (shear stress as a function of shear rate) in linear scales ('up' and 'down' curves): (a) 3.5% starch (continuous lines); 3.5% starch + 0.01% κ-carrageenan (dashed lines); measurement temperature : 15 °C and (b) 4 % starch (continuous lines); 4% starch + 0.01% κ-carrageenan (dashed lines); measurement temperature: 15 °C.

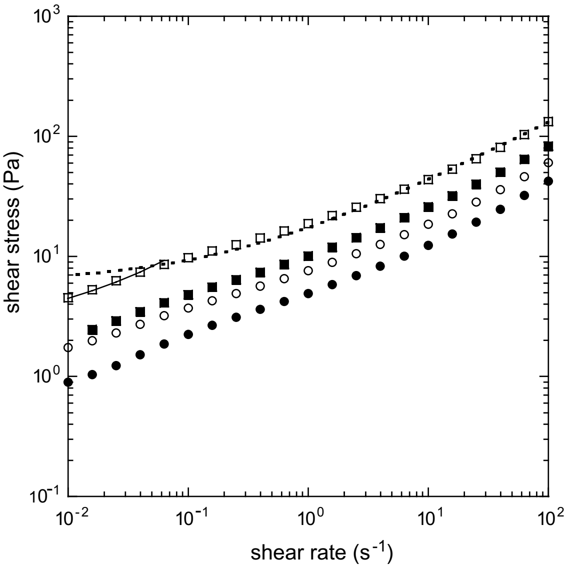

Fig. 5. Flow curves (shear stress as a function of shear rate) in logarithmic scales: 3.5% starch (●); 3.5% starch + 0.01% κ-carrageenan (○); 4% starch (■); 4% starch + 0.01% κ-carrageenan (□); measurement temperature: 15 °C. The dashed line and the continuous line show fits by the Herschell–Bulkley and the Casson equation, respectively (see text).

In Fig. 5, we reported the flow curves plotted in logarithmic scales of the different systems. These curves have been obtained by logarithmically programming the measurements from 100 s^{-1} down to 10^{-2} s^{-1} immediately at the end of the previous measurements. Therefore, these curves reflect the properties of the suspensions after shearing at 100 s^{-1}; they can be considered as equilibrium curves since every experimental point is obtained at equilibrium. The shape of these four curves is comparable. It was interesting to attempt to describe these flow curves by using rheological models. Two equations, that have been already employed for 'custard' sauces, have been compared: the Herschell–Bulkley equation, on one side, and the Casson equation, on the other side.

The Herschell–Bulkley equation is derived from the so-called power-law equation which can be applied when a linear relationship is found in the logarithmic plot. Actually, this condition is only fulfilled when the shear rate range is limited (typically less than two decades). Here the shear rate range was of four decades; it is obvious that the curves were linear only over a limited range, for example from 10 to 100 s^{-1}. The Herschell–Bulkley equation assumes the existence of a yield stress which introduces a deviation from linearity towards the low shear rates. This can be expressed as

$$\tau = \tau_0 + K_1\dot{\gamma}^n$$

with

τ, the shear stress,
τ_0, the yield stress,
$\dot{\gamma}$, the shear rate,
K_1 and 'n' ($n < 1$), parameters of the equation.

The Casson equation also assumes a yield stress; this can be expressed as

$$\sqrt{\tau} = \sqrt{\tau_0} + \eta_P\sqrt{\dot{\gamma}}$$

with

τ_0, the yield stress,
η_P, the plastic viscosity.

An example of the fit is given in Fig. 5 in the case of the 4% starch suspension with 0.01% κ-carrageenan. The fitted curve is shown in dashed lines. Similar results have been obtained for the other suspensions. Clearly, none of both equations was able to describe the flow curves over the entire shear rate range. The Herschell–Bulkley equation appeared suitable for fitting the flow curve from 0.1 s^{-1} up to 100 s^{-1}. The corresponding parameters are given in Table 1. The yield stress was estimated at 6.36 Pa, that is above the shear stress (4.5 Pa) measured at 10^{-2} s^{-1} the lowest shear rate accessed. It is therefore obvious that applying this equation is possible for the highest shear rates (three decades up to 100 s^{-1}) but not towards the low shear rates. Instead, when applying the Casson equation, it was possible to fit the experimental curve only from 10^{-2} to 10^{-1} s^{-1} (continuous line in Fig. 5 for the same example). The shear stress that was estimated by this way (2.25 Pa) could be regarded as more realistic since representative of the behaviour at very low shear rate. The different parameters given by these two equations are given in Table 1.

3.3. Vane rheometry

In Fig. 6, the results obtained using the 'vane' procedure initially employed by Genovese and Rao (2003) in the case of starch-in-water suspensions and then by Tárrega et al. (2006) for custard-type systems are illustrated for two preparations: 4% starch and 4% starch + 0.01% κ-carrageenan. When the sample has not been sheared previously (filled symbols), the shear stress increased steadily up to a maximum and then decreased to a plateau. The initial part of the curve when stress increases corresponds to the elastic response of the material while the decrease is associated to a gradual structure breakdown (Tárrega et al., 2006). It is classically considered that the maximum in the shear stress–time curve can be taken as the yield stress. This has been defined as the static yield stress (τ_{0S}). In contrast, when the sample has been sheared previously at high shear rate (void symbols), there was no maximum. Furthermore, the shear stress was much lower than for the unsheared sample. This can be ascribed to the structure breakdown brought about by the high shear. The shear stress at the plateau has been defined as the dynamic yield stress (τ_{0D}) (Genovese & Rao, 2003). From the comparison between τ_{0D} and τ_{0S} it is possible to estimate the shear sensitivity of the system. The overall results for the different systems are given in Table 1. In the case of 4% starch, τ_{0D} and τ_{0S}, were comparable while τ_{0S} was twice higher than τ_{0D} in the case of 3.5% starch. This latter result means that shearing induced a significant increase in the viscosity. This is reminiscent of the 'antithixotropic' effect that has been reported by several authors in the case of cross-linked starch suspensions (da Silva, Oliveira, & Rao, 1997; Tecante & Doublier, 1999; Tattiyakul & Rao, 2000; Nayouf, Loisel, & Doublier, 2003). This effect is classically ascribed to a rearrangement of the close-packed particles of the suspension, the swollen starch granules in the present case, thus yielding a enhanced organization of the system (Barnes, Hutton, & Walters, 1989).

In contrast, when κ-carrageenan was present, τ_{0S} was much higher than τ_{0D}, which reflects a significant shear sensitivity of the system. This was expected from the flow curves shown in Fig. 4a and b where an hysteresis loop was clearly evidenced for the same systems.

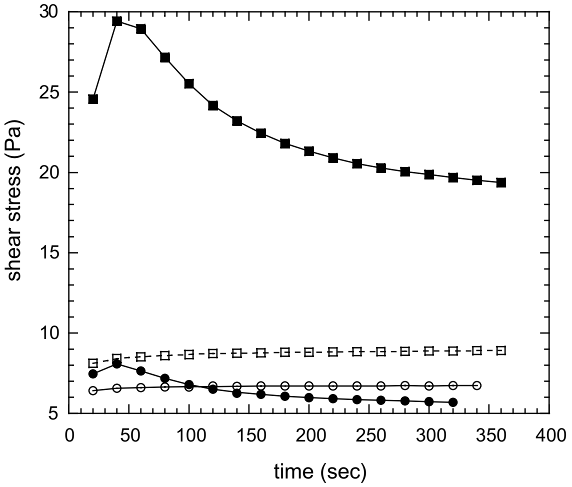

Fig. 6. Shear stress as a function of time using the vane geometry (rotation rate: 0.05 rpm); 4% starch, (●) before shearing and (○) after shearing; 4% starch + 0.01% κ-carrageenan (■) before shearing and (□) after shearing. Measurement temperature: 15 °C.

4. Conclusions

Provided the concentration is high enough, the rheological properties of the dispersions of cross-linked starch in milk display all the features of starch-in-water systems with solid-like properties and a pronounced shear-thinning flow behaviour. These systems can be described as suspensions of close-packed deformable swollen particles. We also can conclude that the rheological properties of a custard-type system are mostly governed by the packing of the swollen starch granules. However, the effect of a very low amount of κ-carrageenan is spectacular by enhancing the viscosity, yielding the 'thixotropic' behaviour and also thermoreversibility.

The main objective of the present study was to compare the rheological information which was obtained using different methods. When performing viscosity measurements, we could describe with good accuracy the flow properties from 10^{-2} to 100 s^{-1}. This can be achieved due to the high sensitivity of the viscometer. The information we can get from these measurements is twofold. We can estimate the extent of the 'thixotropic' behaviour from the hysteresis loop and also the equilibrium flow curves over several decades of shear rates. Using the Herschell–Bulkley equation allowed one to describe the flow curves over three decades. However, the behaviour at low shear rate was poorly described by this equation. The true yield stress could not be estimated by this way. Using the 'vane' method appears quite interesting for the estimation of the yield stress, either at rest (τ_{0S}) or after intense shearing (τ_{0D}). However, we did not describe the entire flow properties by this way. On the other hand, viscoelastic measurements in oscillatory shear provide a way to estimate the properties in non destructive conditions either at rest or after shearing. Different parameters can be obtained : the moduli from measurements in the linearity range (at very low strain amplitude : lower than 1%), on one side, and the critical stress at the G'–G'' cross-over well beyond the linearity limit. This latter parameter, has been found strongly correlated with creaminess (Jellema et al., 2006; de Wijk, Prinz, et al., 2006; de Wijk, Terpstra, et al., 2006). It is worth noting that these values obtained either at rest or after shearing are of the same order of magnitude as τ_{0S} and τ_{0D}, obtained from 'vane' rheometry. Therefore, this may confirm that performing the linearity test beyond the linearity limit can be useful for estimating a parameter related to the yield stress.

From all the results, it appears that 'ideally' at least two methods should be employed for a complete description of the rheological properties of these starchy systems. However, the use of the usual commercial viscometers is possible keeping in mind that the low shear rate range, and particularly the yield stress, cannot be accessed by this way.

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