Tackling slip effects in the non-linear flow properties of gellan fluid gels

Mª Carmen García[a], Sergio Sánchez[a], Luis A. Trujillo-Cayado[a], José Muñoz[a] and Mª Carmen Alfaro[a*]

[a]Universidad de Sevilla, Departamento de Ingeniería Química, 41012, Seville, Spain

Corresponding author: e-mail: alfaro@us.es

Abstract

Gellan gum is a biopolymer widely used in fields such as food, pharmaceutics, chemical or agrochemical. Its property to form strong gel makes possible to produce fluid gels. These materials present an apparent yield stress but its value could be influenced by the wall slip effect on performing the rheological measurements by which it is determined. In this work, the influence of the measuring surface and gap on flow behaviour was first determined. The tests revealed the need to use geometries with rough surfaces although the sample thickness using a parallel plate no influence. Subsequently, the value of yield stress was obtained by means of creep tests (was found to be 4.3 Pa) and, finally, the effect of wall slip on the dynamic viscoelastic behavior was assessed. There was an influence on the extension of the linear viscoelastic region, but not on the viscoelastic functions of the mechanical spectra.

Keywords: Slip effects, Fluids gels, Yield stress, Creep test, Gellan gum

1. Introduction

Several polymers which produce strong gels subject to quiescent cooling such gellan gum[1-5], agar, k-carrageenan and agarose[6-11] can form fluid gels. Such gels are normally obtained on interrupting the formation of strong gels by means of applying shear when the gelation is occurring in polysaccharide solution[12]. These samples exhibit intermediate behaviour between weak and strong gels but are closer to the weak gels.

The essential applications of fluid gels are their capacity as a potential satiety agent[13], as a suspension agent[3] and as an emulsion stabilizer[14].

In this work, fluid gels obtained with low-acyl gellan gum were studied. The native gellan gum is a biopolymer which is commercially produced from Sphingomonas elodea (ATCC 31461) by means of microbial fermentation[3]. Its molecular structure is constituted of a tetrasaccharide repeat unit formed by D-glucose, D-glucuronic and L-rhamnose[15]. This polymer is able to form strong gels under cooling. However, low-acyl gellan gum solutions require cations to produce strong gels. Low-acyl gellan gum fluid gels are aqueous dispersions based on the subsistence of a network consisting of gelled particles[12]. As a consequence of its microstructure, it is necessary to subject the gel to a finite shear stress so that the fluid gels can flow at a significant shear rate. This value of shear stress is the so-called yield stress. Below this stress level, the fluid gel behaves like a solid while above it, the fluid gel behaves like a viscous liquid. The determination of the apparent yield stress is of great importance because its value directly influences the gel flow from a container or the pumping of the fluid gel into a transportation pipeline. Moreover, the application of fluid gels as a suspension agent for particulate matter depends on this yield stress value since the suspension will remain stable if the stress exerted by the action of gravity on the particles is less than the yield stress. For this reason, the main goal of this study has been the determination of apparent yield stress of gellan gum fluid gels.

It is well documented that the yield stress can be determined using different methods. Traditionally it has been obtained by using theoretical models based on the data obtained from flow curve measurements[16]. Recently, various studies have shown other protocols to obtain the yield stress[17,18] and the thixotropy of fluid gels[12,19]. Nevertheless, this measurement of yield stress is not easy because of wall-slip artefacts. Wall slip depletion takes place in the flow of materials which consist of two or more liquid phases in rheometers as a consequence of the dispersed phase being displaced from solid boundaries, resulting in a lower viscosity liquid film. This phenomenon is a consequence of steric, viscoelastic, hydrodynamic, gravitational and chemical forces which act over the dispersed phase directly adjoining the walls[16]. Slip can be shown to occur in rheological measurements when the results using smooth and serrated wall sensors are compared[20]. Therefore, in the current investigation, in order to determine the apparent yield stress of low-acyl gellan gum fluid gels without these wall-slip errors, firstly the influence of different geometries and degrees of roughness of the surface on the flow curves were studied. Moreover, the effect of the gap between the measuring plates, this is to say, the effect of sample thickness on the flow behaviour was also considered. Secondly, once the geometry and appropriate gap for this fluid gel were determined, the yield stress was obtained using two different methods. On the one hand, from creep tests in which a shear stress was instantaneously applied for a predetermined time, and, on the other hand, from flow curves fitted to the Herschel-Bulkley equation.

There are few studies investigating the effect of wall slip phenomena on dynamic viscoelastic behaviour. For this reason, an additional objective of this study consisted of assessing this effect for low acyl gellan gum fluid gels. In order to achieve this purpose, the extension of the linear viscoelastic region by stress sweep measurements at constant frequency was studied as a function of the measuring geometry. Later, frequency sweep tests were carried out within this range so as to ensure that the sample structure would not be damaged by the stress imposed during the measurements. These frequency sweeps were performed with the intention of knowing the influence of the frequency on the storage modulus (G') and the loss modulus (G'') as a function of the geometry used.

Based on the results obtained in this work, a sensor system with a rough surface and a measuring gap of 1000 μm was employed to determine the yield stress by creep tests. Its value was found to be 4.3 Pa. There was an influence of wall slip on the extension of the linear viscoelastic region, but not on the G´ and G´´ of the mechanical spectra.

2. Materials and methods

2.1. Materials

Low Acil gellan (LA gellan), kindly supplied by CP-Kelco (San Diego, USA), was employed. Fluids gels were obtained containing 0.2 wt % gellan gum. In order to obtain Ca^{2+} as a gel-promoting ion, $CaCl_2$ (purity > 98%), supplied by Panreac (Barcelona, Spain), was used. The utilized amount of $CaCl_2$ was 0.15 wt%. To preserve the samples, 0.1 wt % azide sodium (Panreac) was included in the fluid gel formulation. In addition, deionised water was always utilised (electrolytic conductivity: 2.1 mS/cm; calcium concentration: 69.1 ppb; sodium concentration: 16.6 ppb. Room temperature).

2.2. Fluid gel preparation

Gellan gum fluid gels were obtained by the method suggested by Sworn (2009)[3]. LA-gellan gum was slowly added to a vessel placed in a water-bath at 80ºC. The sample was submitted to 700 rpm for 25 min in order to achieve hydration. For this purpose, an Ika-Visc MR-D1 homogeniser (Ika, Germany) and a sawtooth-type impeller were employed. The impeller diameter to the vessel diameter ratio was 0.85. The required temperature was exceeded by 10ºC[21] to ensure perfect gum hydration. Finally, the required amount of $CaCl_2$ was added and the sample was maintained under mechanical treatment at 80ºC.

The quantity of water lost by evaporation was corrected by adding the necessary amounts of water. Gelation was achieved by keeping sample vessel in a water-bath at 20ºC as a coolant. The sample was subjected to mechanical treatment by the Ika-Visc equipment for 1500s to obtain fluid gels. The samples were stored at 4.5ºC for 48 h before performing the rheological study.

2.3. Methods

The rheological tests were performed with an AR-2000 controlled-stress rheometer (TA Instruments, Crawley, United Kingdom). Temperature was set at 20 ºC $\pm$ 0.1 ºC with a Peltier system assisted by a Thermo Scientific circulator. Every test was repeated at least 3 times with a fresh sample and the results are presented as an average.

2.3.1. Determination of the equilibration time

In order to avoid influences of the recent mechanical history after loading the sensor system, the rest time necessary to achieve the equilibrium structure was determined. For this purpose, time sweeps tests were carried out. These measurements were performed with a parallel plate with a rough surface and 60 mm diameter (PP60R) with a measuring gap of 1 mm. Since this test must be performed at shear stress within the linear viscoelastic range (LVR), stress sweeps ranging from $8 \cdot 10^{-3}$ to 10 Pa were carried out previously. Once LVR was determined, time sweeps under oscillatory shear tests at a stress of 0.04 Pa and a frequency of 1 Hz for 60 min were run to estimate the equilibration time. The value obtained as equilibration time was 17 min (data not shown). Therefore, a 17-min rest phase was incorporated for all the rheological tests.

2.3.2. Steady shear flow tests

The measurements were carried out by the controlled-stress rheometer AR2000, in the 0.5-25 Pa shear stress range by a step-wise procedure, with 2 min at each shear stress to reach the steady-state regime. Previously, fluid gels were maintained in the quiescent state at the measuring position for 17 min to permit stress relaxation after the loading procedure. In order to study the existence of wall slip during flow behaviour, the influence of the geometry and measuring gaps on the flow curves were examined. For this, several sensor systems with various geometries were used, namely two parallel plates systems (60 mm diameter), one of them with serrated surface (PP60R) and the other with smooth surface (PP60L), both with a measuring gap of 1000µm, and a cone plate geometry, with measuring gap 32µm and angle 1.059º. In the same way, different measuring gaps (500µm, 750µm and 1000µm) were assessed with the serrated sensor system (PP60R).

2.3.3. Dynamic viscoelastic measurements

In order to study the viscoelastic behaviour, small amplitude oscillatory shear tests were performed. Previously, stress sweeps in a range of $8\cdot10^{-3}$ to 10 Pa at 1 Hz were carried out to determine the linear viscoelastic range (LVR). Once the LVR was determined, frequency sweeps from 3 Hz to 0.01 Hz at 0.02Pa were carried out. With the aim of assessing wall-slip of the sample in both tests, several sensor systems with different geometries (PP60R (measuring gap: 1000 µm), PP60L (measuring gap: 1000 µm) y CP60 (measuring gap: 32µm and angle: 1.059^0) were used.

2.3.4. Creep tests

In these measurements, a constant stress from 1 to 8 Pa was applied for 120 seconds. The sensor system PP60R with measuring gap of 1000 µm was selected to carry out this test. For the reasons explained above, the sample was maintained at rest in the measuring gap and for 17 minutes (the equilibration time) before beginning the test.

3. Results and discussion

3.1. Influence of the surface and geometry of the sensor system on the flow curves

Figure 1 shows the influence of the sensor geometry (PP60R, PP60L and CP60) on the flow curves. As can be observed in Figure 1a, the samples exhibited a shear thinning behaviour. In addition, the results obtained by the parallel plate with serrated surface (PP60R) showed a very shear thinning behaviour, that is to say, at lower shear stress, the apparent viscosity has a tendency to achieve a Newtonian plateau where there is no dependence of shear stress on the viscosity. Additionally, it is possible to observe a sharp decrease in the viscosity by over five orders of magnitude over a small range of shear stress. The values of shear stress at which this change of behaviour takes place is called the yield stress. Not only does Figure 1a show that the flow curve performed with PP60R sensor has a yield stress but the results obtained with this sensor also exhibited higher values of viscosity than those obtained with the other geometries. This fact can be explained by the existence of wall slip phenomena. This also occurred at higher shear stress values but to a lesser extent. Therefore, it can be stated that there was slip and that this was most pronounced at low stress. Similar results were shown in other studies with different systems[22-24].

Figure 1

In Figure 1b, shear stress as a function of shear rate is plotted. Just as in Figure 1a, the very shear thinning behaviour, the existence of a yield stress and wall slip effects can be observed. Moreover, it can be appreciated that the yield stress reached a higher value when the rough sensor was utilized. These results support the need to use a rough geometry in order to avoid the effects produced as a consequence of wall slip.

The experimental data of the shear stress as a function of shear rate has been fitted to the Herschel-Bulkley model:

$$\tau = \tau_0 + k \cdot \dot{\gamma}^n \qquad \text{Eq. 1}$$

Where τ is the stress (Pa) as function of the shear rate ($\dot{\gamma}$), τ_0 is the yield stress (Pa), k is the consistence index ($Pa \cdot s^n$) and n is the flow index.

The fitting parameters to the Herschel-Bulkley model obtained with the different geometries are shown in the Table 1. As can be observed, the higher values of the yield stress and consistence index were achieved when the rough surface was used. This result was a consequence of the wall-slip effect.

Table 1

3.2. Influence of the measuring gap on the flow curves

For these tests, the PP60R sensor system was utilized with different measuring gaps: 500, 750 and 1000μm.

Figure 2a shows that the viscosity was not affected by the measuring gap. Therefore, it can be stated that between 500 and 1000 microns, the measuring gap did not produce a wall-slip effect in the gellan gum fluid gels. The same information can be deduced if Figure 2b is analyzed. This result was supported by the fitting parameter to the Herschel-Bulkley model (Table 2).

Figure 2

Table 2

3.3. Influence of the surface and geometry of the sensor system on the dynamic viscoelastic measurements

In order to evaluate the existence of slippage in the dynamic viscoelastic behaviour of the gellan gum fluid gels, the extension of the linear viscoelastic interval was determined using different sensor systems. Then, the influence of this aspect on the frequency sweep was studied.

A. Determination of the Linear Viscoelastic Region (LVR)

Figure 3 illustrates elastic (G´) and viscous (G´´) moduli as function of the shear stress. The region where both G´ and G´´ remain constant is known as the linear viscoelastic region (LVR) and the value of the shear stress at which they cease to be invariable is called the critical stress (τ_c). As can be observed in Figure 3, the onset of the nonlinear region was accompanied by an increase in the values of G´´. The maximum obtained in G" at the inception of the nonlinear interval could be associated with the energy of dissipation attributed to a microstructural reorganization before the collapse[25]. This fact has been previously detected in some concentrated O / W emulsions and dispersions of surfactants based on laminar liquid crystals[26].

Figure 3

From Figure 3 it can be seen that the LVR obtained with the geometry PP60R was more extensive than those achieved with smooth surface geometries. The values of the critical strain and stress are shown in Table 3. These results support the hypothesis that there were slippage effects.

Table 3

B. Frequency sweeps tests.

These measurements were performed within the LVR. The influence of the frequency on the G´ and G´´ moduli as a function of the sensor geometry used is shown in Figure 4. As can be observed there was no significant influence of the geometry utilized on the mechanical spectra. There was an encouraging agreement between the rough plate-plate, smooth plate-plate and cone-plate geometries. It should be noted that these tests were carried out within a linear viscoelastic region common to all the tested samples. Therefore, it can be stated that while the frequency sweep tests are performed within LVR, the mechanical spectra will not be influenced by the wall slip phenomenon. A similar result was obtained when frequency sweep data for cement paste obtained with a vane are compared with those obtained with smooth-walled concentric cylinders[27].

Figure 4

Additionally Figure 4 showed a) values of G´ over those of G´´ in whole frequency region studied and b) a little influence of the frequency on the viscoelastic modules, especially G´. This result is a typical behaviour of fluid gels.

3.4. Creep measurements

Once the equilibration time, the sensor system and the measuring gap appropriate for this type of samples had been determined, creep tests were carried out in order to obtain the yield stress value.

Figure 5a shows the progress of the capacitance (J) as a function of shear time on applying a nominal constant shear stress of 1 Pa to the sample, which is characterized by very low slope values. The apparent lineal zone achieved at the end of the creep test could lead one to think that the steady state had been obtained. However, a previous work demonstrated the time necessary for this purpose must be over 3600 seconds[28].

A similar behaviour was observed when the test was performed at 4 Pa (Figure 5b). Nevertheless, the results obtained at 4 Pa showed lower reproducibility and a standard deviation lower than those achieved at 1 Pa.

Figure 5

The response of the fluid gels changed drastically under the application of a slightly higher shear stress of 4.3 Pa (Figure 6). A poor reproducibility was observed in these results. Two replicates carried out at 4.3 Pa presented a shape curve similar to those obtained at 1 and 4 Pa (data not shown). However, the other two replicates at 4.3 Pa showed a change in curvature of the capacitance (J) versus shear time involving a decrease in viscosity to achieve the steady state, and it was evident that the fluid gels flowed at a significant shear rate. These results support the fact that this value of shear rate must be consist with the inception of the zone known as "very shear thinning" and, therefore, its value corresponds with a "practical yield stress".

Figure 6

Figure 7 shows the results of imposing 8 Pa on the fluid gellan gum gel. The existence of a curvature can be observed, which revealed the decrease in viscosity and the fact that the fluid gel had started to flow.

Figure 7

3.5. Comparative analysis of the flow curve obtained by creep tests and those obtained by flow tests

From the creep tests it was possible to determine the viscosity at 120 seconds and the associated shear rate for every shear stress applied. In this way, a flow curve can be built and viscosity represented as a function of shear rate in double logarithmic scale (Figure 8a) or as a function of shear stress in double logarithmic scale (Figure 8b). Both figures clearly illustrate the absence of information in at least 4 orders of magnitude of shear rate, which demonstrated the existence of a "very shear thinning behaviour" in these fluid gellan gum gels. This fact was similar to those results obtained from the abovementioned flow tests. However, there was an important difference between both tests. In the flow tests, the result obtained at fixed shear stress was affected by the previous shear history, this is to say, the protocol used. In contrast, in the creep tests, the effect of the shear history was avoided by allowing the sample to relax during the required equilibration time. In addition, fresh sample was used to obtain the results at every shear stress applied.

As mentioned in the introduction, the yield stress can be determined by different methods[17,18]. In this work, it was calculated by means of creep tests. However, it has also been obtained by the fitting of the flow curves to the Herschel-Bulkley model. Comparing the results achieved from both methods, it should be highlighted that the value of yield stress obtained from the fitting equation (3.1 Pa) was clearly lower than that obtained by creep tests (4.3 Pa). Therefore, the value achieved from Herschel-Bulkley model was underestimated. This fact can be attributed to the mechanical history to which the fluid gellan gum gel was submitted during the flow curves.

4. Conclusion

The use of several geometries of different surface roughness brought about different results in flow curves. Flow curves reveal the occurrence of wall slip when smooth surfaces are used and they support the conclusion that the PP60R was the most appropriate sensor system to obtain them. In addition, these flow curves demonstrated that the fluid gels of gellan gum exhibited a very shear thinning behaviour. On the other hand, no effect on the flow curves was observed as result of the use of different measuring gaps when PP60R was utilized as the sensor system. With regard to LVR, it was found also was influenced by wall slip. The extent of the LVR was reduced by the employment of smooth surfaces. However, mechanical spectra were not affected by wall slip since the measurements were carried out within the common linear viscoelastic range. These tests indicated this sample showed a weak gel behaviour characterized by G´ values above G´´ values with low frequency dependence.

Creep tests made it possible to determine the yield stress value, which turned out to be 4.3 Pa. From these tests a flow curve without the effect of mechanical history can be obtained.

Finally, from comparison of the two different methods used to determine the yield stress (fitting the flow curve to Herschel-Bulkley’s model and creep tests), it can be concluded that creep is the most suitable rheological measurement.

5. Acknowledgments

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Figure captions

Figure 1. Influence of the sensor system geometry and surface on the flow curves of fluid gels containing 0.2 wt % low acyl gellan gum and 0.15 wt % $CaCl_2$. a)Viscosity vs. shear stress, b) Shear stress vs. shear rate. Temperature: 20ºC.

Figure 2. Influence of the measuring gap on the flow curves of fluid gels containing 0.2 wt % low acyl gellan gum and 0.15 wt % $CaCl_2$. a)Viscosity vs. shear stress, b) Shear stress vs. shear rate. Sensor PP60R. Temperature: 20ºC.

Figure 3. Influence of the sensor system geometry and surface on the determination of the linear viscoelastic region at 1 Hz of the fluid gels containing 0.2 wt % low acyl gellan gum and 0.15 wt% $CaCl_2$. Temperature: 20ºC.

Figure 4. Influence of the sensor system geometry and surface on the mechanical spectra of the fluid gels containing 0.2 wt % low acyl gellan gum and 0.15 wt % $CaCl_2$. Temperature: 20ºC.

Figure 5. Capacitance (J) as a function of the shear time in creep tests of fluid gels containing 0.2 wt % low acyl gellan gum and 0.15 wt % $CaCl_2$. a) At 1 Pa. b) At 4 Pa Temperature: 20ºC.

Figure 6. Capacitance (J) as a function of the shear time in creep tests of fluid gels containing 0.2 wt % low acyl gellan gum and 0.15 wt % $CaCl_2$. Average of three replicates and standard deviation. Temperature: 20ºC.

Figure 7. Capacitance (J) as a function of the shear time in creep tests at 8 Pa of fluid gels containing 0.2 wt % low acyl gellan gum and 0.15 wt % $CaCl_2$. Temperature: 20ºC.

Figure 8. Flow curve obtained from creep measurement results of fluid gels containing 0.2 wt % low acyl gellan gum and 0.15 wt % $CaCl_2$. a) Viscosity vs. shear rate, b) Viscosity vs. shear stress. Temperature: 20ºC.

Tables.

***Table 1*. Influence of the sensor system geometry and surface on the fitting parameters to the Herschel-Bulkley model. Temperature: 20ºC.**

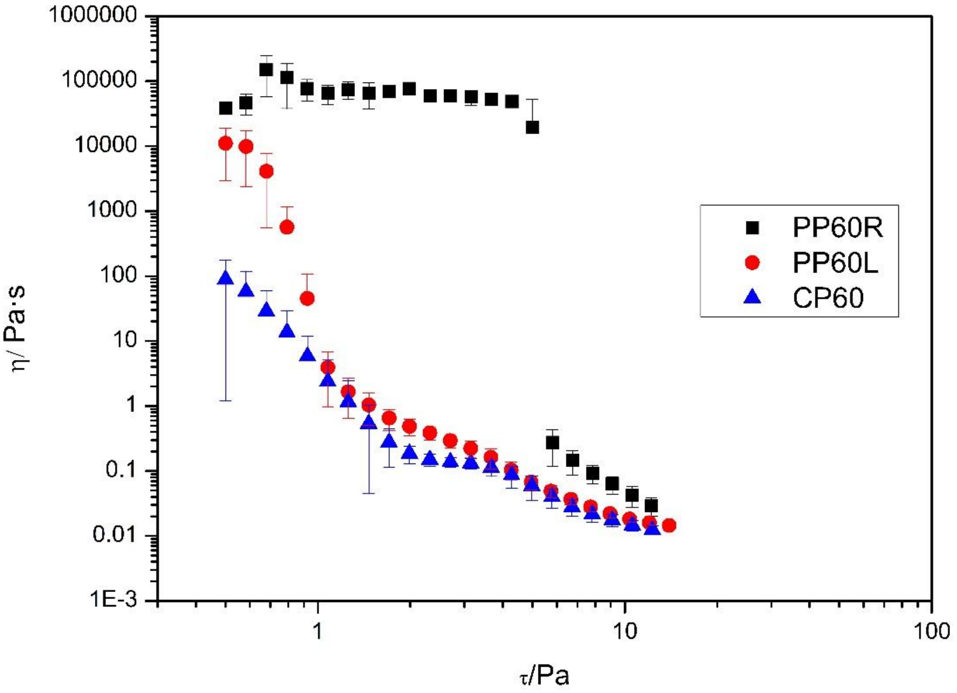
Sensor system	$\tau_0 \pm SD$ (Pa)	$k \pm SD$ ($Pa \cdot s^n$)	$n \pm SD$
PP60R	3.10±0.27	0.71±0.21	0.43±0.05
CP60	0.47±0.12	0.66±0.07	0.41±0.02
PP60L	1.68±0,26	0.33±0.08	0.53±0.036

***Table 2*. Influence of the measuring gap on the fitting parameters to the Herschel-Bulkley model. Temperature: 20^{0}C.**

Measuring gap (μm)	***τ±SD (Pa)***	***k±SD (Pa·s^n)***	***n±SD***
500	3.66±0.29	0.49±0.06	0.47±0.03
750	3.30±0.32	0.58±0.24	0.45±0.07
1000	3.11±0.27	0.70±0.21	0.43±0.05

***Table 3*. Influence of the sensor system geometry and surface on the determination of the linear viscoelastic region at 1 Hz. Temperature: 20^{0}C.**

Sensor system	**Replicados**	τ_c***(Pa)***	γ_c
PP60R	Mean value	0.15	0.005
	SD	0.02	4E-04
CP60	Media	0,03	8E-04
	SD	0,01	1E-04
PP60L	Media	0,08	2E-03
	SD	0,02	4E-04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1000
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10
1
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τ/Pa
PP60R
PP60L
CP60

Figure 1a

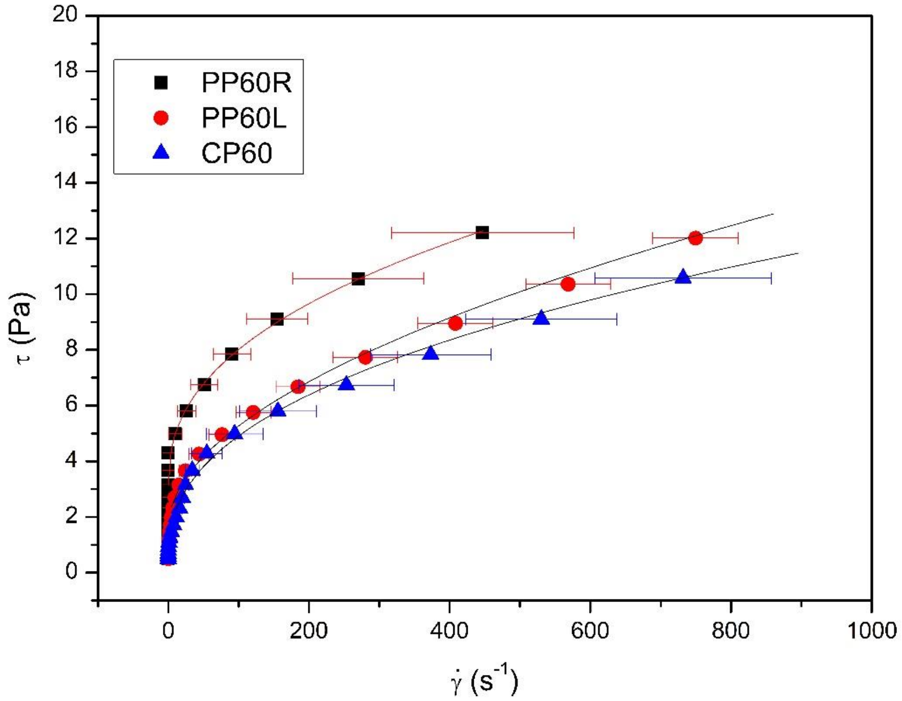

Figure 1b

Figure 1. Influence of the sensor system geometry and surface on the flow curves of fluid gels containing 0.2 wt % low acyl gellan gum and 0.15 wt % $CaCl_2$. a)Viscosity vs. shear stress, b) Shear stress vs. shear rate. Temperature: 20ºC.

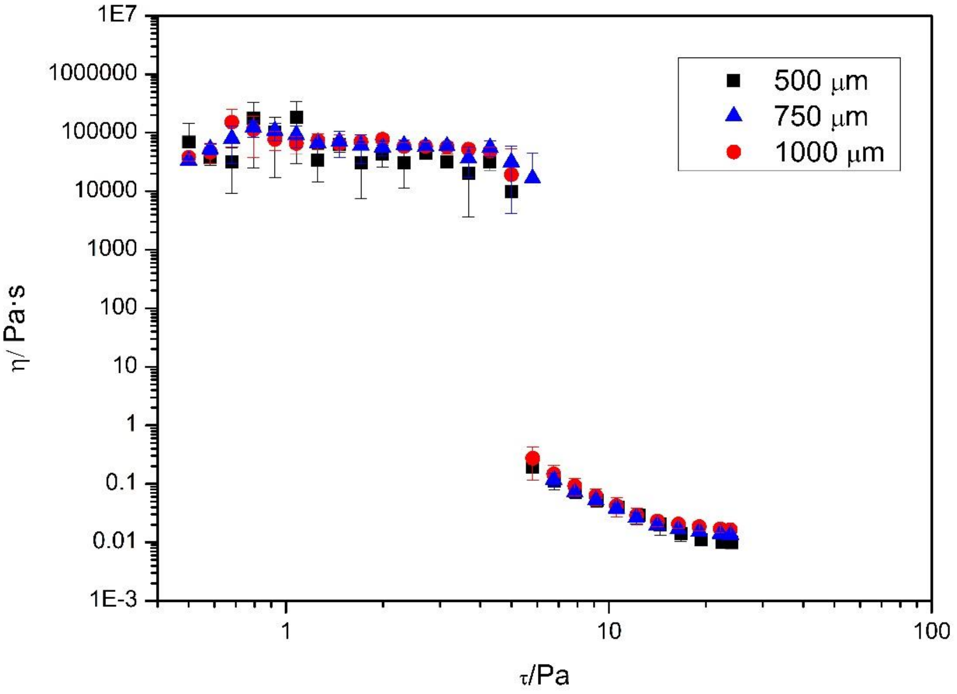

Figure 2a

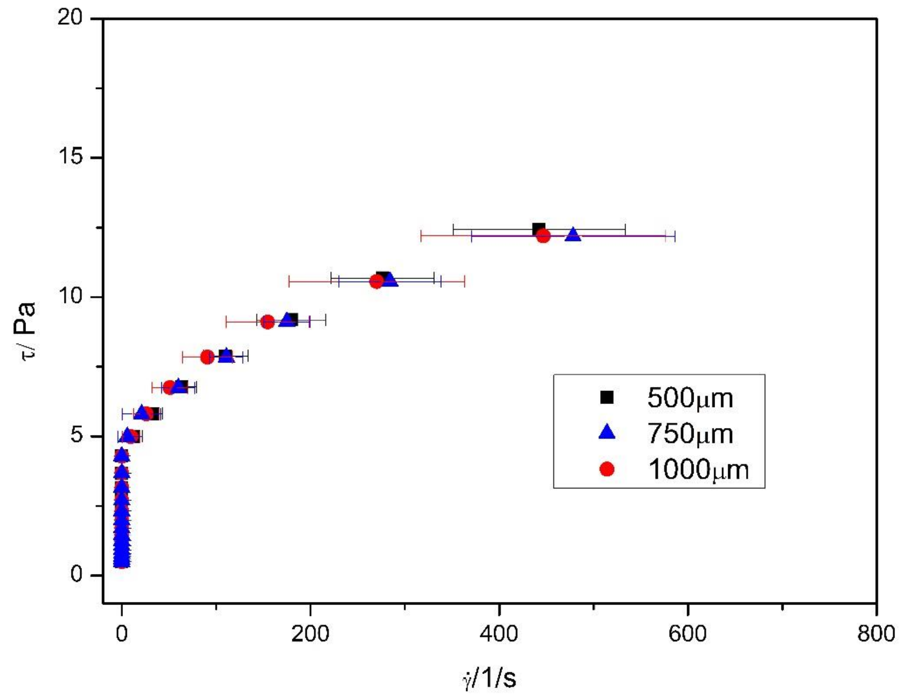

Figure 2b

Figure 2. Influence of the measuring gap on the flow curves of fluid gels containing 0.2 wt % low acyl gellan gum and 0.15 wt % $CaCl_2$. a)Viscosity vs. shear stress, b) Shear stress vs. shear rate. Sensor PP60R. Temperature: 20ºC.

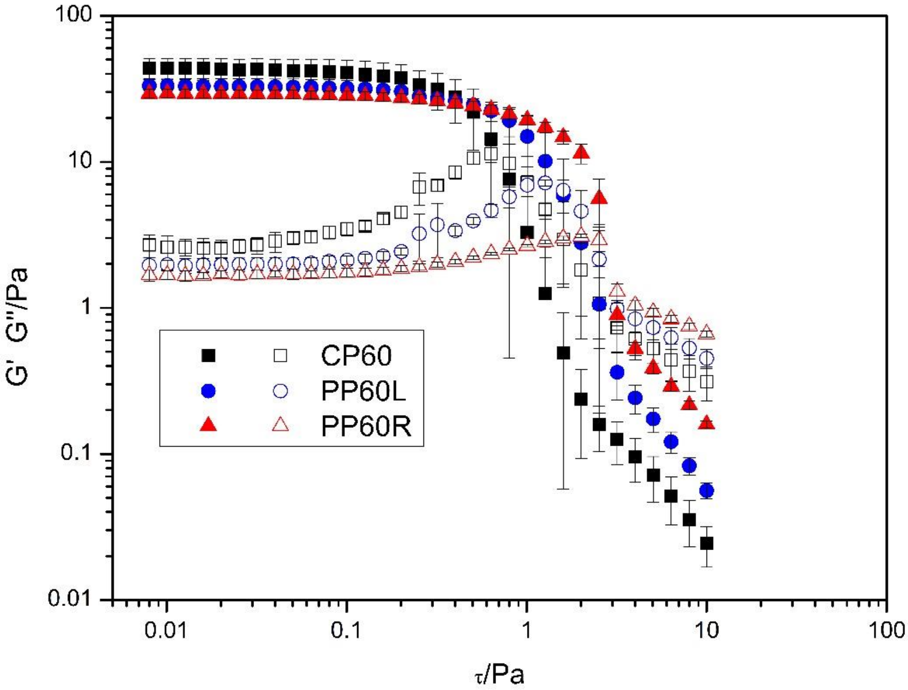

Figure 3. Influence of the sensor system geometry and surface on the determination of the linear viscoelastic region at 1 Hz of the fluid gels containing 0.2 wt % low acyl gellan gum and 0.15 wt% $CaCl_2$. Temperature: 20ºC.

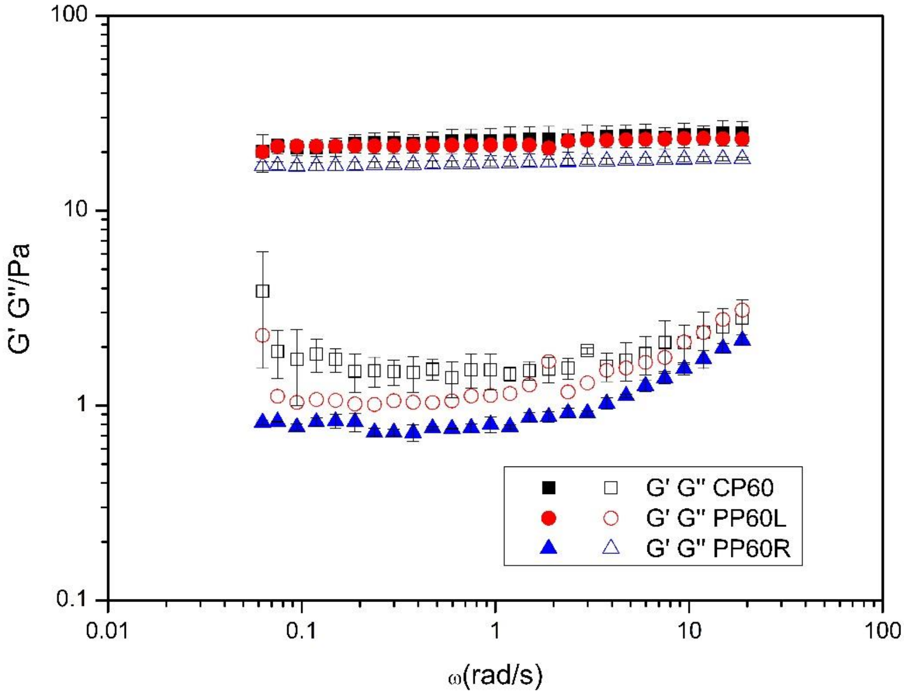

Figure 4. Influence of the sensor system geometry and surface on the mechanical spectra of the fluid gels containing 0.2 wt % low acyl gellan gum and 0.15 wt % $CaCl_2$. Temperature: 20ºC.

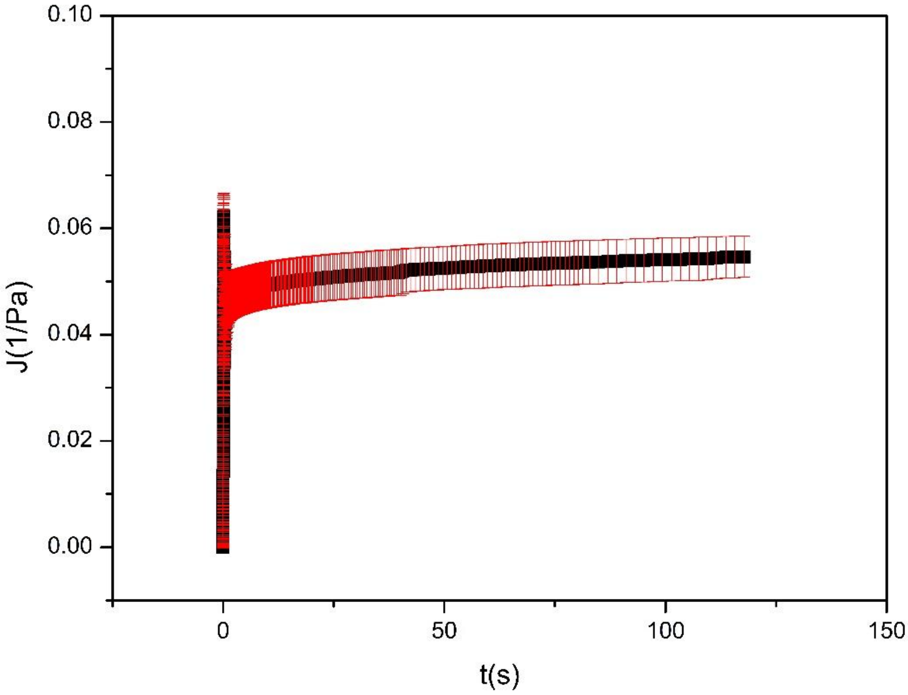

Figure 5a

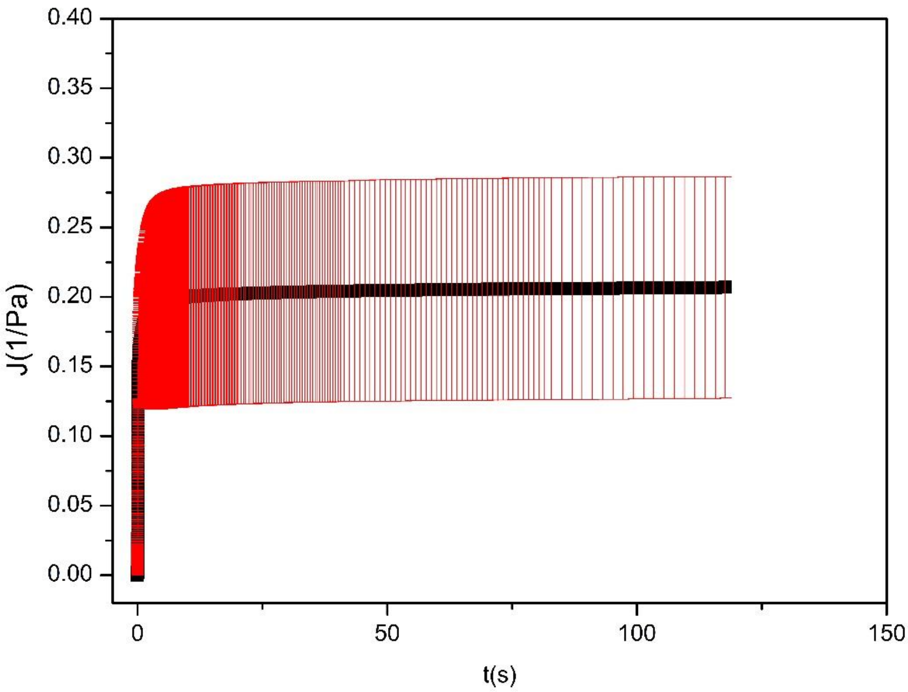

Figure 5b

Figure 5. Capacitance (J) as a function of the shear time in creep tests of fluid gels containing 0.2 wt % low acyl gellan gum and 0.15 wt % $CaCl_2$. a) At 1 Pa. b) At 4 Pa Temperature: 20ºC.

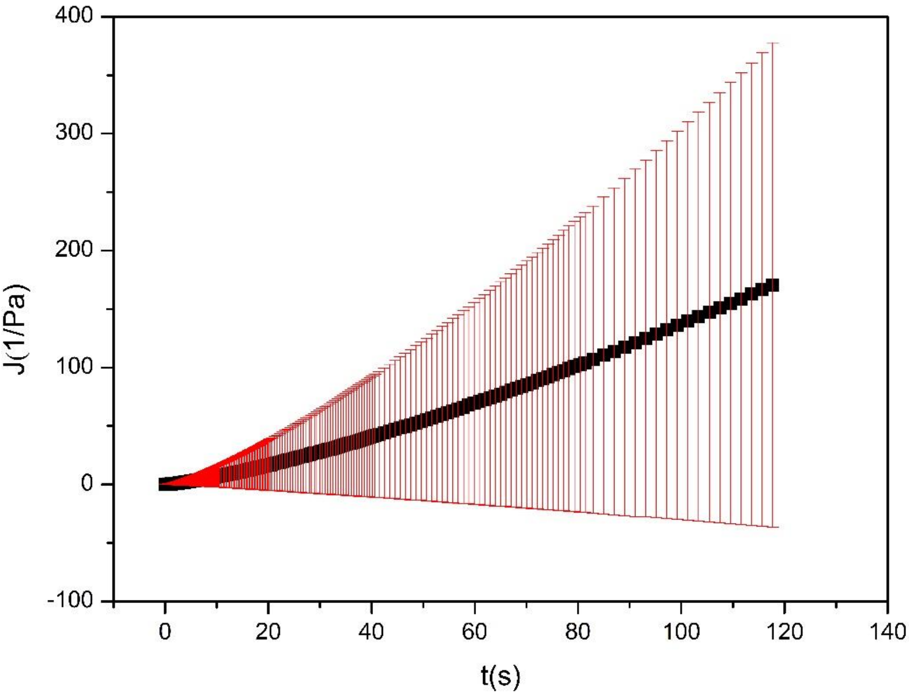

Figure 6. Capacitance (J) as a function of the shear time in creep tests of fluid gels containing 0.2 wt % low acyl gellan gum and 0.15 wt % $CaCl_2$. Average of three replicates and standard deviation. Temperature: 20ºC.

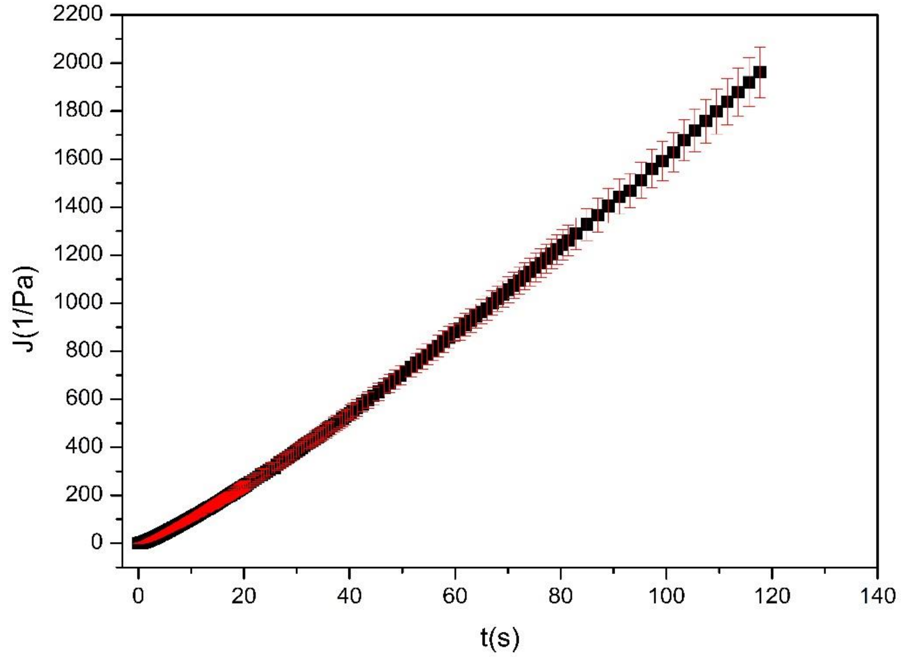

Figure 7. Capacitance (J) as a function of the shear time in creep tests at 8 Pa of fluid gels containing 0.2 wt % low acyl gellan gum and 0.15 wt % $CaCl_2$. Temperature: 20ºC.

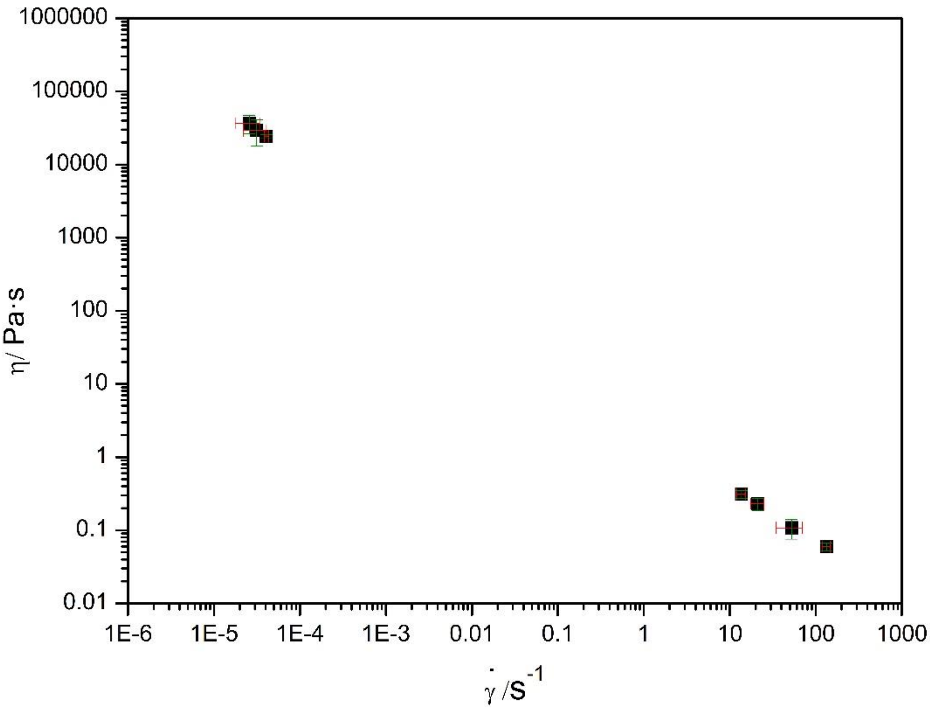

Figure 8a

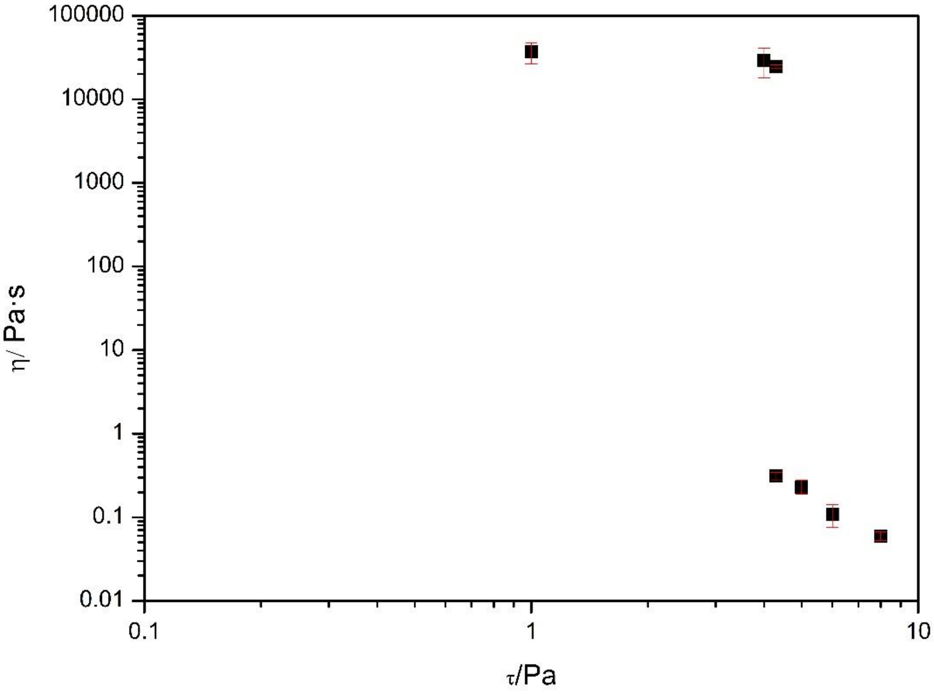

Figure 8b

Figure 8. Flow curve obtained from creep measurement results of fluid gels containing 0.2 wt % low acyl gellan gum and 0.15 wt % $CaCl_2$. a) Viscosity vs. shear rate, b) Viscosity vs. shear stress. Temperature: 20ºC.