## 1 Flow, dynamic viscoelastic and creep properties of a biological polymer

# 2 produced by Sphingomonas sp. as affected by concentration

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#### 9 Abstract

In this work, the influence of the concentration on the flow behaviour, dynamic 10 viscoelastic and creep properties of diutan gum in aqueous solution was investigated. 11 Diutan gum is a biopolymer which belongs to the sphingans group. To know its 12 rheological properties and its microstructure as a function of the concentration is directly 13 related to the current and future applications of this biological polymer. Mechanical 14 spectra showed a crossover point between G' and G" which changed as a function of 15 diutan gum concentration. A master curve for the frequency dependence on the  $\eta^*$  was 16 obtained. The creep compliance results made it possible to deduce the yield stress value 17 and they were fitted to Burgers model. A shear-thinning behaviour was exhibited by 18 diutan gum aqueous solutions, which was fitted to the Carreau-Yasuda model. Higher 19 G', G",  $\tau_0$  and  $\eta_0$  values and lower  $\omega_c$ ,  $J_e^0$ ,  $\dot{\gamma}_c$  and n values were obtained by increasing 20 the gum concentration, it is being possible to modulate the viscoelasticity, viscosity and 21 22 shear resistance as a function of concentration. A more complex structure with stronger

- entanglements between macromolecules of diutan was obtained when the concentrationof diutan increases.
- 25

#### 26 Keywords

- 27 Diutan gum, Rheology, Viscoelasticity, Creep compliance, Yield stress
- 28

# 29 1.- Introduction

Diutan gum is an anionic biopolymer whose backbone structure consists of a repeated 30 units of D-glucose, D-glucoronic acid, D-glucose and L-rhamnose) and its molecular 31 weight ranges from 2.88 to 5.18 MDalton [1, 2]. It is obtained from Sphigomonas sp. 32 ATCC53159 by aerobic fermentation and it belongs, therefore, to the sphingans group 33 34 [3]. As a consequence of its capability to modify the rheological properties of the aqueous solutions, nowadays it has applications in various fields. Long Xu et al. [4] reported the 35 effect of temperature and salinity on the rheological properties of diutan gum solutions 36 from  $10^{-3}$  wt% to 0.4 wt% concentration range. In this work [4], the authors concluded 37 that there was no influence of the temperature and salinity conditions on the physical 38 properties of these biopolymer solutions. This fact makes the use of diutan gum very 39 40 interesting in applications where the environmental conditions could be extremely harsh, such as in the case of tertiary petroleum recovery [4]. Furthermore, diutan gum can be 41 42 used as a viscosity-enhancing admixture in concrete formulation. In the concrete industry, these admixtures are usually used to improve the rheological properties of materials based 43 on cement, principally the viscosity. In this way, the grouts containing diutan gum exhibit 44 45 a shear thinning behaviour, and as a consequence, the mixtures show high viscosity at 46 low shear, presenting an apparent Newtonian viscosity, whereas the fluidity at high shear47 rate improves due to a decrease in the viscosity [5].

48 Furthermore, recently Carmona et al. [6] studied the effect of shear and temperature on49 the microstructure of 0.5 wt% diutan gum aqueous solutions.

Nevertheless, the literature concerning diutan gum is scarce and there is not an exhaustive
study of the influence of diutan gum concentration on its linear viscoelastic and flow
properties, as it is done in this work.

From an engineering point of view, the study of the steady state properties of materials is 53 most rewarding. However, numerous materials exhibit an elastic behaviour along with 54 55 their steady state properties, and therefore, in order to characterize them rheologically, it is necessary to use a method which studies the combination of elastic and viscous 56 behaviours of materials. Unsteady state tests supply a method to study viscoelastic 57 substances, and for this purpose, transient and oscillatory measurements are the most used 58 tests. In this work, creep compliance measurements have been carried out to obtain 59 60 information that can be useful in assessing phenomena taking place in industrial processes. In these tests, the sample is submitted to a constant shear stress for a certain 61 time and the evolution of the strain is observed. Data obtained from these experiments 62 63 can be related to industrial applications influenced by the effects of gravity, such as sedimentation or levelling [7]. In addition, knowledge concerning creep compliance could 64 65 be helpful in the study of construction and building materials. For this purpose, several researchers have used this test in order to predict the thermal cracking of asphalt concrete 66 used in flexible pavements [8, 9, 10]. 67

To get better insights into the properties and microstructure of the diutan gum dispersions,
the influence of its concentration (0.1,0.2,0.3,0.4 and 0.5 wt%) on the viscoelastic linear

- and flow properties been evaluated in this paper by means of small amplitude oscillatory
  shear tests, creep compliance measurements and the figures of flow curves.
- 72 **2. Materials y Methods**
- 73 *2.1. Materials*

Diutan gum of type KELCO-VISTM DG kindly supplied by CP Kelco (San Diego, USA) 74 has been used. The diutan gum was studied in the 0.1-0.5 wt % concentration range and 75 0.5 wt % NaCl, provided by Panreac, was employed as source of cations. This 76 77 concentration range was selected because the most relevant property of the biopolymers is their capability to modify the flow behaviour in aqueous solutions at low 78 79 concentrations. The concentrations studied are similar to those used in other biopolymers 80 such xanthan gum or gellan gum [11-14]. In order to preserve the diutan gum solution, 0.1 wt % sodium azide (Panreac) was introduced into the formulation. In addition, water 81 82 milli-Q was utilized. Each system studied was denoted as MX, being X the wt % of diutan gum contained in the sample. 83

- 84 2.2. *Methods*
- 85

#### 2.2.1. Diutan gum solution preparation.

Batches of 600 g of diutan gum solution were prepared. For this purpose, diutan gum powder was added to a vessel containing NaCl, sodium azide and milli-Q water. The mixture was stirred constantly at 700 rpm for 5 hours by means of an Ika-Visc MR-D1 homogenizer (Ika, Germany) and a coulex impeller. The diameter ratio between the impeller and vessel was 0.85. The diutan gum solution was then submitted to a temperature of 80°C for one hour while stirring at 700 rpm was maintained using the same equipment. Finally, the water lost by evaporation was replaced.

93 Samples were kept at 4.5°C for at least 48 hours before their characterization.

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# 2.2.2. Rheological measurements

All rheological measurements, except flow curves, were performed by means of an AR-2000 controlled-stress rheometer (TA Instruments, Crawley, United Kingdom) assisted by a sensor system consisting of a rough parallel plate with a diameter of 60 mm (PP60R) and a measuring gap of 1 mm. Temperature was set at 25 °C  $\pm$  0.1 °C with a Peltier system aided by a Thermo Scientific circulator.

Flow curves were determined with a Haake-Mars controlled-stress rheometer (Thermo
Scientific, Karlsruhe, Germany) connected to a Phoenix circulator (Thermo-Scientific) at
25°C. Measurements were carried out using, as a sensor system, a coaxial cylinder (inner
diameter 20.0 mm, outer diameter 21.7 mm, cylinder length 30.0 mm) (Z20) with a treated
surface.

106 *2.2.2.1. Time sweep tests* 

In order to avoid any effects of the mechanical history of the samples after placing them
the sensor system, the equilibration time was determined. For this purpose, time sweeps
at 1 Hz and 0.5 Pa were performed for 2 hours at 25°C.

Equilibration times were obtained using a first-kinetic equation which has been used to
fit the G´ values in recovery systems such as suspensions with xanthan gum as stabilizer
[15].

113 
$$G' = G_0' + (G_{\infty}' - G_0)[1 - \exp(-k \cdot t)^m]$$
(1)

where G<sup> $\prime$ </sup> is the elastic component, G<sub>0</sub><sup> $\prime$ </sup> is the G<sup> $\prime$ </sup> value for zero recovery time obtained from the equation (1), G<sub>∞</sub><sup> $\prime$ </sup> is the G<sup> $\prime$ </sup> value for a complete structural recovery, k is a kinetic 116 coefficient and m is a fitting parameter indicating the kinetic order which has been made117 equal to 1 in this particular case.

The values of equilibration time were in the 1000-2900 s range (data not shown).
However, to prevent samples from drying, it was decided to apply an equilibration time
of 900 s in all systems studied.

121 2.2.2.2. Determination of linear viscoelastic range (LVR)

Before carrying out mechanical spectra studies stress sweep tests were performed, ranging from  $8 \cdot 10^{-3}$  to 10 Pa at 3 different frequencies (0.1, 1 and 3 Hz) in order to determine the linear viscoelastic range.

125 2.2.2.3. Frequency sweep test

Once the linear viscoelastic region was established, frequency sweep tests wereperformed in the 15-0.05 rad/s range at constant shear stress within LVR.

128 *2.2.2.4. Time sweep tests* 

Oscillatory shear time tests were used to complete the mechanical spectra at frequency values below 0.5 rad/s. These measurements were performed in the  $5.5 \cdot 10^{-3}$ -  $8 \cdot 10^{-3}$  rad/s range at fixed shear stress values, which guaranteed the LVR.

132 2.2.2.5. Creep compliance tests

133 Creep compliance measurements were performed while maintaining a constant stress,134 which ranged from 0.01Pa to 2.5Pa for 2 hours.

In order to model the viscoelastic behaviour of the sample studied, creep compliance results have been fitted to Burgers model. This model consists of Maxwell and Kelvin elements connected in series. To obtain a better representation of data, in Burgers model two Kelvin elements have been included. Therefore, Burgers model was described as:

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$$J = \left(\frac{1}{G_0}\right) + \left(\frac{1}{G_1}\right) \left(1 - \exp(-\frac{t}{\lambda_{ret1}})\right) + \left(\frac{1}{G_2}\right) \left(1 - \exp(-\frac{t}{\lambda_{ret2}})\right) + \frac{t}{\eta_0}$$
(2)

140 Where J is the compliance as a function of time, t,  $\frac{1}{G_0}$  is equal to J<sub>0</sub> which is instantaneous

141 compliance, at t=0,  $\frac{1}{G_1} = J_1$ ;  $\frac{1}{G_2} = J_2$ ,  $\lambda_{\text{ret}}$  is the retard time which is the time needed to

reach maximum deformation,  $\eta_0$  is the Newtonian viscosity in the steady state. The two exponential elements of equation (2) represent viscoelastic behaviour described by the two Kelvin elements. In this way,  $\frac{1}{G_0} = J_0$  is the purely elastic response, and  $t/\eta_0$  is the

145 Newtonian response when the steady state is reached. In the measurements carried out in 146 the linear viscoelastic range, the steady state compliance,  $J_e^0$ , is equal to the following 147 equation [7].

148 
$$J_e^0 = J_0 + J_1 + J_2$$
 (3)

In addition, when the linear portion of the creep curves is fitted to a linear equation, it is possible to determine the value of  $J_e^0$  from the interception and  $1/\eta_0$  from the curve slope.

## 151 2.2.2.6. Steady shear flow tests

The measurements were performed in the 2.0 - 15 Pa shear stress range by a step-wise procedure, with 30 points and 300s at each shear stress to reach the steady-state regime. In all rheological tests, in order to prevent the effects of mechanical history, the sample was kept in the measuring gap and in a quiescent state for the equilibration time of 900 seconds to permit stress relaxation before beginning the test. Every measurement was

157 performed at least 4 times on fresh samples.

ANOVA ONE WAY method has been employed to determine the occurrence of
significant differences among the obtained results. In order to fulfill this purpose,
OriginPro 8 software was used.

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## 163 **3. Results and discussion**

#### 164 *3.1. Determination of linear viscoelastic range (LVR)*

Stress sweep tests were performed at 0.1Hz, 1 Hz and 3 Hz to estimate the linear 165 166 viscoelastic range (LVR). Firstly, elastic component (G<sup>'</sup>) values above the viscous component (G') values were observed in each sample studied (data not shown). In 167 addition, as the stress increases two regions can be clearly differentiated: a) the so-called 168 169 linear viscoelastic region with constant values of elastic modulus and viscous modulus and b) a non-linear region where both viscoelastic functions start to decrease. It should 170 be noted that the inception of the non-linear response was identified by a slight fall in G<sup>-</sup> 171 172 accompanied by a clear decrease in G<sup>''</sup>.

The value of stress from which G' and G" values are no longer constant is known as the critical stress,  $\tau_c$ , and it corresponds to the stress that leads to the first non-linear changes in the structure [16]. Its value indicates the degree of association between macromolecules [17]. The ANOVA ONE study demonstrated regardless of the frequency studied, by increasing the concentration of diutan gum aqueous solutions, the values of  $\tau_c$  increase (Table 1), indicating the entanglements between the macromolecules in solution are stronger and, therefore, the shear resistance.

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181 *3.2. Frequency sweep tests* 

Mechanical spectra demonstrated (Figure 1) that the diutan gum solutions containing 0.5 182 183 wt % NaCl exhibited viscoelastic properties at all concentrations studied. In order to avoid 184 the drying of M0.5 sample, the data at smaller frequencies were obtained by means of time sweep tests. In the mechanical spectra of all samples there was a predominance of 185 the elastic component, G', over the viscous component, G'', at higher frequencies. 186 However, a crosspoint was observed at low frequency,  $\omega_c$ , which progressively shifted to 187 188 lower frequencies with increasing gum concentration (Table 2) as supported the ANOVA ONE WAY study. The crossover point is inversely related to the terminal relaxation time 189 of the samples,  $\lambda$ , so that, as the concentration of diutan gum in the sample increases, the 190 191 time necessary for its structure to relax is greater. The behaviour observed can be attributed to the formation of a network or incipient gel based on a limited number of 192 193 entanglements, characterized by long relaxation times. As the gum concentration 194 decreases, the degree of entanglements decreases as well as the relaxation time. Therefore, we observed a change in behaviour from more solid-like viscoelastic 195 196 properties to more marked liquid-like viscoelastic properties by decreasing the gum 197 concentration.

In order to normalize the analysis, a master curve for the frequency dependence on the complex viscosity at the reference concentration of 0.3 wt % was obtained (Figure 2). As can be observed in Figure 2, the results exhibited an acceptable superposition in the whole range of frequencies studied. Similar superpositions have been found with other biopolymers but using G' and G" rather than  $\eta^*$  [18, 19].

The values of vertical and horizontal shift factors are shown in Table 2. Interestingly, the vertical shift factor ( $F_{\eta*}$ ) decreases by increasing the gum concentration. Conversely, the horizontal shift factor,  $F_{\omega c}$ , increases. The concentration dependence of this last factor can be described by a power law,  $F_{\omega c} \propto c^{3.3}$  (R<sup>2</sup>=0.95). Cuvelier and Launay [ 20, 21] for xanthan solutions, which also belongs to the sphingans group, above the overlap concentration found an stronger concentration dependence ( $c^{7.4}$ ).

209 Creep compliance tests

210 Figure 3 exhibits the creep compliance test results at all shear stresses applied for the sample containing 0.2 wt % diutan gum. In Figure 3, the test data has been expressed in 211 terms of the creep compliance function, which is J (t) =  $\gamma/\tau$ , where  $\gamma$  is the shear strain 212 and  $\tau$  is the constant shear stress applied. As can be observed, compliance curves obtained 213 214 in the range of shear stress applied from 0.05Pa to 0.1 Pa overlap as a consequence of carrying out the tests at shear stresses within the viscoelastic linear range [6]. An increase 215 216 in shear stress value of 0.4Pa provoked the inception of non-linear viscoelastic behaviour 217 of the sample. Similar results were obtained when testing the other samples studied.

Figure 4 shows the steady state compliance,  $J_e^0$ , and Newtonian viscosity,  $\eta_0$ , as a function 218 of the shear stress applied for all diutan gum concentrations studied. This figure displays 219 a significant decrease of the  $J_e^0$  and  $\eta_0$  values from a critical value of shear stress. In 220 221 addition, the fitting parameters of Burgers model (equation (2)) did not fulfill the linear 222 condition expressed by equation (3) for shear stress values higher than the critical shear 223 stresses. For these reasons, it could be deduced that the inception of the non-linear viscoelastic range has been reached and, therefore, those shear stress values could be 224 225 considered to be the yield stress values ( $\tau_0$ ). That is, the shear stress value which has to 226 be exceeded so that the sample can flow at significant values of shear rate. This fact 227 reveals the possibility of determining the yield stress by creep compliance tests. In recent 228 studies, the yield stresses of materials with very shear thinning behaviour were obtained 229 by creep-recovery-creep measurements [12, 13]. The yield stress values for samples M0.2, M0.3, M0.4 and M0.5 are respectively 0.1Pa, 0.25Pa, 0.5Pa and 0.7Pa. As can be observed, there was an increase in yield stress values as a function of the diutan gum concentration, which is in agreement with the results obtained in oscillatory tests. The more structured the sample, the higher the stress threshold to initiate the flow. These results were supported by ANOVA ONE study.

Additionally, an increase in the  $\eta_o$  values and a decrease in  $J_e^0$  values with diutan gum concentration can be observed in Figure 4. These results are in agreement with the occurrence of a stronger viscoelastic structure by increasing the concentration of gum in solution and, consequently, with a structure more resistant to flow, as was observed from oscillatory measurements.

## 240 *3.3.Steady shear flow tests*

Figure 5 shows flow curves of the samples studied (full symbols). Also included in this figure are the viscosity values obtained by means of creep compliance tests. In this way, viscosities values at exceptionally small shear rate values in which the time needed to achieve steady state would be extremely large are incorporated. These results have been shown in Figure 5 as open symbols.

A shear-thinning behaviour was exhibited for all systems studied, which is typical of polymeric molecules aligned in the flow direction as the shear rate increases. As a consequence of this, a decrease in the polymeric interactions between adjacent chains occurs. This behaviour was fitted to the Carreau-Yasuda model (equation (4) [22]:

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251 
$$\eta_{=}\eta_{\infty} + \frac{\eta_{0} - \eta_{\infty}}{\left(1 + \left(\frac{\dot{\gamma}}{\dot{\gamma}_{c}}\right)^{p}\right)^{\left[\frac{1-n}{p}\right]}}$$
(4)

Where  $\eta_0$  is the zero shear viscosity,  $\eta_\infty$  is infinite shear viscosity,  $\dot{\gamma}_c$  is critical shear rate related to the inception of structural collapse, n is the index flow and p is a fitting parameter.

255 The fitting parameters shown in Table 4 indicate that, as the gum concentration increased, 256 the viscosity value increased. Similar behaviour was observed in other gums such as 257 xanthan gum, Sterculia apetala gum or Prosopis juliflora gum [17, 23]. Higher concentration of gum improves the entanglements of molecular chains and therefore 258 259 increases the viscosity. A typical behaviour was found for the critical shear rate,  $\dot{\gamma}_c$ , since it decreased with diutan gum concentration. As expected, the lowest  $\dot{\gamma}_c$  was shown by the 260 most concentrated dispersion since this parameter is related to the longest relaxation time 261 262 of the system. It should be remarked that the longer the terminal relaxation time, the higher the solid-like viscoelastic properties exhibited by the sample. The flow index, n, 263 264 showed a tendency to decrease as the gum concentration was increased on account of the increasing non-Newtonian properties caused by the greater structural complexity of gum 265 dispersions. 266

267

#### 268 **4.** Conclusions

269 This work presents a study of linear viscoelastic properties and flow behaviour of diutan gum solutions in concentrations ranging from 0.1 wt % to 0.5 wt %. All of them showed 270 a linear viscoelastic range which increased with the diutan gum concentration. In addition, 271 mechanical spectra exhibited G' values above G'' and a crosspoint between both 272 functions at low frequency, which was a function of the diutan gum concentration. The 273 274 crossover frequency, related to the relaxation time, decreased as the diutan gum 275 concentration increased, indicating that the sample structure needs a longer time to relax as its concentration increases. Moreover, a superposition of the mechanical spectra in 276

term of complex viscosity as a function of the frequency demonstrated that the samplescontaining from 0.1 wt % to 0.5 wt % diutan gum showed a similar structure.

279 On another hand, creep compliance data were well fitted to the Burgers model. The fitting parameters and the plot of creep compliance function made it possible to determine the 280 281 onset of the non-linear viscoelastic region for all the systems studied, and this is related to the yield stress value. This value of shear stress increased with the diutan gum 282 concentration. Additionally, these tests, along with flow curves, made it clear that these 283 284 samples presented a shear-thinning behaviour. Subsequently, the viscosity values determined from creep tests at low shear stress were used to complete the flow curves in 285 order to obtain more real values of the zero-shear viscosity. The final flow curves 286 287 achieved from creep measurements and flow curves fitted well to the Carreau-Yasuda 288 model. The fitting parameters obtained supported those obtained from the creep compliance test. Zero-shear viscosity increased with the diutan gum concentration. 289 290 Consequently, the critical shear rate and flow index showed smaller values as the gum 291 concentration increased.

All rheological results are in agreement with the occurrence of a sample structure more resistance to shear and flow which has higher viscoelastic properties and viscosity with increasing concentration of diutan gum. Therefore, It is possible to modulate the rheological properties of diutan gum aqueous solutions modifying its concentration. Results obtained are very interesting for industrial applications of diutan gum not only as viscosity enhancer but also as stabilizer agent. Furthermore, these applications can be developed at very low concentrations.

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#### Tables

- Table 1. Determination of dynamic linear viscoelastic region at studied frequencies (0.1, 1 and 3 Hz.

	τ <sub>с</sub> (Ра) <sub>0.1 нz</sub>	τ <sub>c</sub> (Pa) <sub>1 Hz</sub>	τ <sub>с</sub> (Ра) <sub>з нz</sub>
M0.1	0.21±0.03	0.24±0.02	1.30±0.42
M0.2	0.68±0.19	0.67±0.07	0.20±0.01
M0.3	1.41±0.27	1.84±0.23	1.79±0.42
M0.4	2.31±0.44	> 3	2.72±0.26
M0.5	> 3	> 3	>3

## 

Table 2. Determination of crossover points and vertical and horizontal shift factors to get the master curve at studied diutan gum concentrations. T=25°C. 

	$\omega_{c}$ (rad/s)	λ (s)	G´=G´´(Pa)	Fωc	η* <sub>corte</sub> (Pa·s)	F <sub>η*</sub>
M0.1	$\textbf{0.340} \pm \textbf{0.010}$	2.94	0.20±0.01	0.12	0.82	41.00
M0.2	0.085±0.003	11.8	0.53±0.01	0.45	8.75	3.82
M0.3	0.039±0.001	27.9	0.96±0.02	1	33.42	1
M0.4	0.017±0.002	58.8	1.26±0.07	2.30	106.9	0.31
M0.5	0.006±0.002	166.7	1.49±0.09	5.75	331.2	0.09

Table 3. Fitting parameters of the Carreau-Yasuda equation for diutan gum solutions at

-		η∞	ηo	SDη₀	γ <sub>c</sub> (s <sup>-1</sup> )	SDγ <sub>c</sub>	р	SDp	n	SDn	R <sup>2</sup>
		(Pa∙s)	(Pa·s)	(Pa∙s)		(s⁻¹)					
-	M0.5	10-3	2528.6	174.4	0.0015	7·10 <sup>-4</sup>	0.52	0.10	0.11	-	0.998
-	M0.4	10-4	1106.3	33.4	0.0013	4·10 <sup>-4</sup>	0.95	-	0.17	-	0.998
	M0.3	10-4	281.0	8.05	0.005	7·10 <sup>-4</sup>	0.65	-	0.17	-	0.999
	M0.2	10-4	44.5	0.08	0.018	2·10 <sup>-4</sup>	0.85	-	0.18	5·10 <sup>-3</sup>	0.997
-	M0.1	2.2.10-4	3.72	0.20	0.07	5·10 <sup>-3</sup>	1.5	-	0.21	0.01	0.993

382 studied concentrations. SD stands for standard error of fitting parameters.

383

## **Table captions.**

Table 1. Determination of dynamic linear viscoelastic region at studied frequencies (0.1,1 and 3 Hz.

Table 2. Determination of crossover points and vertical and horizontal shift factors to get
 the master curve at studied diutan gum concentrations. T=25°C.

Table 3. Fitting parameters of the Carreau-Yasuda equation for diutan gum solutions at
studied concentrations. SD stands for standard error of fitting parameters.

391

## 392 Figure Captions

- 393 Figure 1. Influence of diutan gum solution on the mechanical spectra. It is included the
- points obtained from frequency sweep tests (close symbols) and those obtained from time
- sweep tests (open symbols). T=25°C.
- Figure 2. Master curve for the frequency dependence on the complex viscosity taking as
- the reference the sample containing 0.3 wt % diutan gum. T=25°C.
- 398 Figure 3. Creep compliance test results at all shear stresses applied for the sample
- 399 containing 0.2 wt % diutan gum. T=25°C.
- 400 Figure 4. Influence of diutan gum solution on  $J_e^0$  (A) values and  $\eta_o$  values (B). T=25°C.

401 Figure 5. Influence of diutan gum solution on the viscosity values obtained from flow
402 curve measurements (close symbols) and creep compliance tests (open symbols).
403 T=25°C.

404



Figure 1. Influence of diutan gum solution on the mechanical spectra. It is included the points obtained from frequency sweep tests (close symbols) and those obtained from time sweep tests (open symbols). T=25°C.



Figure 2. Master curve for the frequency dependence on the complex viscosity taking as the reference the sample containing 0.3 wt % diutan gum.  $T=25^{\circ}C$ .



Figure 3. Creep compliance test results at all shear stresses applied for the sample containing 0.2 wt % diutan gum. T=25°C.



Figure 4. Influence of diutan gum solution on  $J_e^0$  (A) values and  $\eta_o$  values (B). T=25°C.



Figure 5. Influence of diutan gum solution on the viscosity values obtained from flow curve measurements (close symbols) and creep compliance tests (open symbols).  $T=25^{\circ}C$ .