

1 **Effect of temperature and shear on the microstructure of a microbial**  
2 **polysaccharide secreted by Sphingomonas species in aqueous solution.**

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9 **Abstract**

10 Diutan gum is a biological polymer produced by Sphingomonas sp. In aqueous  
11 solution it shows gel-like structure under quiescent conditions. However, the  
12 flow-induced evolution of its viscoelastic properties and its microstructure are  
13 not known. In this work, the viscoelastic moduli were obtained under a flow field,  
14 applied in parallel, as a function of the temperature for 0.5 wt% diutan gum  
15 aqueous solutions. As both stress and temperature increase a decrease in the  
16 viscoelastic properties occurred, due to the fact that the molecular interactions  
17 decreased. Nevertheless, at stresses within the zero-shear viscosity region of  
18 the flow curves, no changes were observed. In addition, high stress values  
19 dampened the effect of the temperature. The results obtained are very  
20 interesting from an industrial application perspective.

21 **Key words:** Diutan gum, Parallel superposition, Creep, Viscoelasticity.

22

23 **1. Introduction**

24 Polysaccharides are biological macromolecules whose structures consist of a  
25 great number of monosaccharide units connected to each other by O-glycosidic  
26 linkages. They are considered to be biopolymers since they are obtained from  
27 natural sources, such as plants, bacteria, mammals, mollusks and fungi. Their  
28 physical properties, such as solubility, interface properties or flow behaviour

29 are a direct consequence of the polysaccharide structure which is dependent  
30 on the monosaccharides present in the macromolecule [1]. They exhibit a high  
31 added value and their main applications are as foam and emulsion stabilizers,  
32 gelling and thickening agents, and in water binding or avoidance of ice  
33 recrystallization or syneresis [2].

34 In this work, diutan gum was used, which is obtained by bacterial secretion from  
35 the *Sphingomonas* sp genus like other biopolysaccharides such as gellan,  
36 rhamsan or welan gums. For this reason, they are called sphingans [3-5]. The  
37 outstanding functions of sphingans make their use extremely interesting in an  
38 extensive number of biotechnological applications involving personal care,  
39 pharmaceuticals, oilfields, food and production processes [5,6]. Diutan gum is  
40 an anionic biopolymer whose structure consists of a configuration of  $\beta$ -D-  
41 glucose,  $\beta$ -D-glucuronic acid and  $\alpha$ -L-rhamnose units whose molecular weight  
42 is about 2.88 to 5.18 million Daltons [7,8]. Diutan gum aqueous solutions exhibit  
43 viscoelastic properties and shear thinning behaviour at low concentration [9].  
44 These properties make possible the use of the diutan gum as a thickener,  
45 stabilizer, emulsifier and suspending or binding agent. It is widely used in the  
46 concrete industry [10] as a viscosity-enhancing admixture and in ternary oil  
47 recovery. In some of these applications, diutan gum is submitted to extreme  
48 conditions of both temperature and shear. Xu et al. [9] found that diutan gum  
49 aqueous solutions at very low concentration, unlike the most biopolymers, show  
50 gel structure which remains practically unaltered with temperature and salts in  
51 the range from 298 k and 348 k. Nevertheless, the evolution of the  
52 viscoelasticity and, therefore, of the microstructure of diutan gum under flow is  
53 still unknown. For this reason, along with the fact that current rheometers make  
54 it possible to carry out these measurements easily, a deep study of diutan gum  
55 microstructure under flow is of great interest since the information obtained may  
56 be useful from an industrial point of view. One way of proceeding would be to  
57 use a parallel superposition. This test consists of the superposition of oscillatory  
58 shear to a principal steady state shear in the same direction and at the same  
59 time. This measurement provides the mechanical spectra of the sample when  
60 the steady state has been achieved at a certain shear stress. Performing this  
61 procedure at different shear stresses not only makes it possible to observe the  
62 evolution of the microstructure with the shear stress applied and the time of

63 application, but also to compare it with that deduced from the mechanical  
64 spectra obtained under quiescent conditions [11].

65 In order to gain a deeper knowledge concerning diutan gum microstructure and  
66 its future possible applications, small amplitude oscillatory shear  
67 measurements, creep compliance tests, steady state tests and superimposed  
68 oscillatory-on-steady-state shear tests were performed on 0.5 wt % diutan gum  
69 solution containing 0.5 wt % NaCl and 0.1 wt % sodium azide at different  
70 temperatures (10°C, 15°C, 20°C, 30°C and 40°C).

71

## 72 **2. Materials and Methods**

### 73 *2.1. Preparation of diutan gum aqueous solutions*

74 Diutan gum aqueous solutions were prepared at a concentration of 0.5 wt%.  
75 Firstly, the dispersions were stirred at room temperature for 3 hours. Then, they  
76 were heated at 70°C for 45 min and subsequently they were cooled to room  
77 temperature. The cooling rate was about 1°C/min. Finally the water lost due to  
78 evaporation was replaced and 0.5 wt% NaCl and 0.1 wt% [12] sodium azide  
79 were added.

80 .

### 81 *2.2. Rheological Tests*

82 After preparation, the sample was allowed to equilibrate for 24 hours before  
83 starting the rheological characterization. All measurements were performed by  
84 means of a controlled stress rheometer, DHR3 (TA Instruments) with a rough  
85 surface sensor, PP40R (40 mm of plate diameter). All rheological tests were  
86 carried out at constant temperature (10 °C, 15 °C, 20 °C, 30 °C or 40 °C). At  
87 higher measuring temperatures, the samples were loaded into the sensor  
88 system at 20°C instead of the test temperature, in order to avoid loading the  
89 sample on a hot surface. Then, they were heated up to the measuring  
90 temperature by a Peltier system. Equilibration time prior to rheological tests was  
91 determined by small amplitude oscillatory shear (SAOS) experiments at 1Hz  
92 frequency as a function of time and it was estimated as 300 s.

93 Frequency sweeps in SAOS experiments in a range of 30-0.1 rad/s were  
94 carried out by selecting a stress (0.1 Pa) within the linear range previously  
95 determined by stress sweeps at a 1 Hz fixed frequency.

96 Creep experiments on the diutan gum solutions were performed by applying a  
97 constant shear stress for 1800 s when these stresses were 0.1-2 Pa or for 900s  
98 when the stresses ranged from 3.5 Pa to 12 Pa.

99 Shear flow measurements were carried out using a stress control multistep  
100 protocol from 0.05 to 25 Pa.

101 In experiments of superimposed oscillatory-on-steady shear, first, the sample  
102 was sheared for 15 or 30 min by means of a creep test, in order to ensure that  
103 the steady state was achieved. Then, a steady-state shear flow and an  
104 oscillatory perturbation were applied in the same direction. The stresses of  
105 added shear ranged from 0.1 to 12 Pa. It should be noted that previously the  
106 region of linear viscoelasticity was established and a stress within this region  
107 was chosen as the oscillatory operating stress. With regard to the pre-shear,  
108 note that below 2 Pa the required time was 30 min and above 3.5 Pa this time  
109 was 15 min. Superposition measurements were performed at 10, 15, 20, 30 and  
110 40 °C.

111 All rheological tests were performed using a solvent trap in order to avoid water  
112 loss by evaporation and they were carried out in duplicate.

### 113 **3. Results and Discussion**

#### 114 *3.1. Mechanical Spectra without superposition of steady shear*

115 Figure 1 illustrates the changes in  $G'$  and  $G''$  as a function of the frequency at  
116 10 °C, 15 °C, 20 °C, 30 °C and 40 °C.

117  Figure 1.

118 Diutan gum aqueous solutions showed typical weak-gel behaviour, with elastic  
119 modulus values,  $G'$ , always above loss modulus,  $G''$ , in the overall frequency  
120 range studied, without there being a crossover point between both viscoelastic  
121 functions. Therefore, it can be stated that the diutan gum solutions had a  
122 character that was more solid than liquid and that the deformations are

123 essentially elastic and recoverable. This rheological behaviour is characteristic  
124 of systems with a high degree of internal rearrangements and it is similar to that  
125 presented by other hydrocolloids, such as welan gum or xanthan [13,14].

126 No influence of an increase of temperature from 10 °C to 40 °C on the  
127 viscoelastic moduli was observed. Additionally, it was found that  $G'$  was always  
128 higher than  $G''$  regardless of the temperature. This result revealed that these  
129 solutions behaved as a weak gel even at high temperatures and that their  
130 structure, in practice, was not sensitive to changes of temperature within the  
131 investigated temperature range (10°C to 40°C). According Xu et al. [9] this  
132 behaviour can be attributed to the fact that the diutan gum tends to form a  
133 perfect double helix able to retain water molecules in its core, which causes this  
134 insensitivity to temperature.

### 135 *3.2. Creep Tests*

136 In Figure 2 the compliance (J) versus time as a function of shear stress at 20 °C  
137 and 40 °C of diutan gum aqueous solutions is shown.

#### 138 Figure 2

139 Regardless of the temperature, similar compliance values were obtained at  
140 similar shear stress. As can be observed in Figure 2 the limit of the linear  
141 viscoelastic zone was around 2-3.5 Pa. In addition, a linear zone towards the  
142 end of test was attained. From this region, both a viscosity value and the shear  
143 rate associated for each shear stress applied can be calculated. The flow curve,  
144 at 20 °C, built from creep tests, is shown in Figure 3, by way of example.

#### 146 Figure 3

147  
148 Additionally, creep measurements made it possible to know the time required to  
149 reach the steady state at every stress applied. For this purpose, shear rate  
150 versus time was plotted, making it clear that above the linear viscoelastic range  
151 ( $\sigma > 2$  Pa) a time of 15 min was sufficient to attain the mentioned steady state.

### 153 *3.3. Steady Shear Flow*

#### 154 Figure 4

155 Steady shear flow curves of diutan gum aqueous solutions at different  
156 temperatures and at 0.5 wt% are shown in Figure 4. This figure illustrates a  
157 trend to reach a Newtonian viscosity at low shear rates, the so-called zero  
158 shear viscosity ( $\eta_0$ ). In this region, the rates of formation and breakdown of  
159 macromolecular entanglements are similar. For this reason, the viscosity  
160 remains constant. The value of the zero shear viscosity can be related to the  
161 density of entanglements among macromolecules. Therefore, the high viscosity  
162 shown by diutan gum can be attributed to this cause, namely a high intertwining  
163 of their chains. From the critical shear rate, a decrease in the viscosity was  
164 observed with an increase in the shear rate. The decrease in the apparent  
165 viscosity with the increase in the shear rate is due to the fact the shear deforms  
166 and disaggregates the entanglements, causing their alignment in the flow  
167 direction which reduces the hydrodynamic drag and, therefore, the viscosity  
168 [15]. Generally, at high shear rates there is a tendency to reach another  
169 Newtonian plateau ( $\eta_\infty$ ), but in this study a complete alignment of diutan gum  
170 macromolecules was not observed.

171 Two points should be noted. On the one hand, the underestimated  $\eta_0$  value  
172 obtained from flow tests compared with that obtained from creep tests. This  
173 result may be attributed to the fact that in flow tests the experimental data at  
174 fixed stress were affected by the protocol used, namely the mechanical history.  
175 In contrast, in creep tests this does not happen. On the other hand, the good  
176 agreement between the values of viscosity obtained from both tests in the shear  
177 thinning region. By way of example, in Figure 3 results of creep and flow tests at  
178 20 °C are plotted.

179 In all cases, the Carreau model [16] was used to fit the experimental data:

180

181 
$$\eta = \frac{\eta_0}{(1 + (\dot{\gamma}/\dot{\gamma}_c)^2)^{\frac{1-n}{2}}} \quad (Eq. 1)$$

182 Where  $\eta$  is the apparent viscosity (Pa s),  $\eta_0$  is the zero-shear rate viscosity  
183 (Pas),  $\dot{\gamma}_c$  is the critical shear rate ( $s^{-1}$ ) and  $n$  is the power law exponent. It should

184 be noted that to carry out the fit of experimental data the  $\eta_0$  values calculated  
185 from creep experiments were used.

186 Table 1 shows the fitting parameters and quality. The flow index,  $n$ , which is  
187 related to the slope ( $n-1$ ) of the shear thinning region, always showed the same  
188 value, regardless of the temperature considered. The  $n$  values less than 1 were  
189 consistent with a shear thinning behaviour. It is important to draw attention to  
190 the fact that a marked pseudoplastic behaviour is of great interest because it  
191 facilitates pumping [17]. Guar gum exhibited similar behaviour with a constant  
192 flow index value (0.31) from 5 °C to 65 °C[18].

193 Table 1

194 With regards to the onset shear rate for shear thinning behaviour ( $\dot{\gamma}_c$ ), after an  
195 initial increase up to 20°C, an apparent plateau was observed. It should be  
196 remarked the great experimental difficulty involved in obtaining the Newtonian  
197 plateau at low shear rates which, in complex fluids as is the case in many  
198 concentrated polysaccharide solutions, requires a long test time in order to  
199 reach the steady state.

200 The decrease of  $\eta_0$  with temperature was fitted to the so-called modified  
201 Andrade's equation [19] (Figure 5):

202

$$203 \quad \ln \frac{\eta}{\eta_{ref}} = \frac{E_a}{R} \left[ \frac{1}{T} - \frac{1}{T_{ref}} \right] \quad (Eq. 2)$$

204 where,  $\eta$  is the viscosity (Pa s),  $\eta_{ref}$  is the viscosity at a reference temperature  
205 (Pa·s),  $E_a$  is the activation energy for the flow process (J/mol),  $R$  is the  
206 universal gas constant (8,34 J/mol),  $T$  is the absolute temperature (K) and  $T_{ref}$  is  
207 the reference absolute temperature (293 K).

208 Figure 5

209 The  $E_a$  value obtained in the zero-shear rate Newtonian region (in Andrade's  
210 equation,  $\eta = \eta_0$ ) was  $10.6 \pm 0.1$  KJ/mol. It is worth noting the low value of this  
211 parameter, which indicated that the diutan gum aqueous solutions were not very  
212 sensitive to temperature and the microstructure was not very vulnerable to  
213 change. To better understand the low value of this  $E_a$ , note that the activation  
214 energy at  $1 \text{ s}^{-1}$  of the exopolysaccharide obtained by microbial fermentation of

215 Pseudomonas oleovorans turned out to be  $35.5 \pm 2.4$  kJ / mol [20]. Note also  
216 that at zero shear rate  $E_a$  should be greater, since higher lifetimes for  
217 entanglements between polymer chains are necessary, thus more energy is  
218 required to promote the flow. Other authors [9] obtained  $E_a$  of diutan gum (2.58  
219 J/mol) and the results were 3 times lower than that obtained in this work, which  
220 may be attributed to the lower gum concentration. In any case, flow curve  
221 results indicated a high thermal resistance of the diutan gum.

222

### 223 *3.4. Steady shear superimposed in parallel on an oscillatory shear flow*

224 As mentioned above, before starting the parallel superposition measurements,  
225 the linear viscoelastic region was determined. In Figure 6, the critical stress  
226 and strain versus the stress applied were plotted at 20°C. A trend to a decrease  
227 in the critical stress with increasing the stress applied was found at all  
228 temperatures studied. From 10 °C to 40 °C the linear viscoelastic zone  
229 remained unchanged.

230

Figure 6

231 Figure 7 shows the dependence of parallel storage modulus,  $G'_{//}$ , and parallel  
232 loss modulus,  $G''_{//}$ , on the frequency of pre-sheared diutan gum aqueous  
233 solutions. It is worth noting that at stresses within the plateau zone of the flow  
234 tests (1.2 and 2 Pa) no changes were observed in the values of the parallel  
235 modules, matching the behaviour found at zero stress. In contrast, at higher  
236 stresses a decrease in the magnitude of the parallel storage modulus with  
237 increasing stress applied and the occurrence of a crossover point between both  
238 moduli were observed. A similar behaviour to that obtained with these stresses  
239 was previously reported for polymer solutions [21], although in these works the  
240 parallel viscous modulus also decreased with the applied shear rate. In  
241 contrast, in this investigation  $G''_{//}$  remained invariable up to 6 Pa. Above this  
242 stress, its value decreased and its dependence on frequency seriously  
243 increased. This behaviour was due to the fact that by increasing the stress the  
244 rearrangement of diutan molecules in the flow direction decreased the friction,  
245 and thus, the viscosity and also decreased the interactions between polymer



246 chains so the slope of both parallel moduli increased. The higher the stress the  
247 higher slope and the more liquid the behaviour.

248 In Figure 7B, results at 40 °C are shown. As can be observed a similar trend to  
249 that found at 20 °C was produced.

250 **Figure 7**

251 From the previous curves, the terminal relaxation time,  $\lambda$ , of the diutan gum  
252 aqueous solutions was calculated as the inverse of the crossover frequency ( $\omega_c$ )  
253 in rad/s units. It was then plotted against the shear stress applied as a function  
254 of temperature (Figure 8). As expected, as the shear stress or the temperature  
255 increased, a decrease in  $\lambda$  occurred due to a decrease in the connectivity of the  
256 macromolecules as result of a decrease in molecular interactions. Additionally,  
257 as observed in the flow curves, Figure 8 illustrated that the shear (increase of  
258 stress) dampened the effect of temperature.

259 **Figure 8**

260

#### 261 **4. Conclusions**

262 Diutan gum aqueous solutions at 0.5 wt% exhibit a typical weak-gel behaviour  
263 with  $G'$  values always higher than  $G''$  regardless of the temperature. Under flow,  
264 they show a shear thinning behaviour which was fitted to the Carreau model.  $\eta_0$   
265 obtained from creep tests were higher than those obtained from flow curves,  
266 indicating the relevance of the mechanical history on their values. The  
267 dependence of  $\eta_0$  on the temperature was fitted to the Andrade equation, giving  
268 a value of activation energy for the flow process of 10.6 kJ/mol. When the flow  
269 is superimposed on the oscillatory test, regardless of the temperature, the  
270 parallel moduli do not change at stresses below 2 Pa, their values being similar  
271 to those obtained under quiescent conditions. Both temperature and stress  
272 decrease the molecular interactions and, therefore, the viscoelasticity. In  
273 addition, at the highest stresses the effect of the shear dampened the influence  
274 of the temperature. The information obtained from these results is very  
275 interesting from an industrial application perspective.

276

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281

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372 **Tables**

373

374 **Table 1.** Carreau model fitting parameters for 0.5 wt % diutan gum solution  
375 as a function of temperature in the (10–40) °C range ( $R^2 > 0.99$ ).

T(°C)	$\eta_0$ (Pa s)	$\dot{\gamma}_c$ (s <sup>-1</sup> )	n	R <sup>2</sup>
10	1633±70	$8.93 \times 10^{-04} \pm 2.1 \times 10^{-05}$	0.380±0.021	0.990
15	1452±40	$1.10 \times 10^{-03} \pm 1.7 \times 10^{-05}$	0.363±0.017	0.994
20	1359±14	$1.34 \times 10^{-03} \pm 9 \times 10^{-05}$	0.357±0.003	0.994
30	1347±50	$9.92 \times 10^{-04} \pm 1.2 \times 10^{-05}$	0.394±0.015	0.992
40	1049±30	$1.24 \times 10^{-03} \pm 2.4 \times 10^{-04}$	0.386±0.014	0.993

376

377 **Figure captions**

378

379 **Figure 1.** Mechanical spectra for 0.5 wt % diutan gum solution as a function of  
380 temperature in the (10–40) °C range. Standard deviation for G', G'' < 5%.

381

382 **Figure 2.** Compliance as a function of time for various shear stress. Results  
383 obtained by creep compliance tests for a 0.5 wt. % diutan gum solution at 20°C  
384 and 40°C. Standard deviation for J < 10%.

385

386 **Figure 3.** Shear rate dependence of steady state viscosity obtained by  
387 combining creep compliance tests (closed symbols) and multistep flow curves  
388 (open symbol) for 0.5 wt % diutan gum solution at 20°C. Standard deviation for  
389  $\eta < 10\%$ .

390

391 **Figure 4.** Steady shear flow curves for 0.5 wt % diutan gum solution as a  
392 function of temperature in the (10–40) °C range. Standard deviation for  $\eta <$   
393 10%.

394

395 **Figure 5.** Arrhenius-type plot for the temperature dependence of the zero-shear  
396 viscosity for 0.5 wt % diutan gum solution. The line shows data fitting to the  
397 Andrade equation.

398

399 **Figure 6.** Critical stress and strain values for a 0.5 % (m/m) diutan gum solution  
400 as a function of parallel shear stress at 20°C. Error bars correspond to standard  
401 deviation from the one-way analysis of variance (ANOVA) at a 5% significance  
402 level.

403

404 **Figure 7.** Parallel storage moduli  $G_{II}'$  and parallel loss moduli  $G_{II}''$  as a function  
405 of angular frequency for various parallel shear stress values. For a 0.5 % (m/m)  
406 diutan gum solution at 20°C and 40°C. Standard deviation for  $G_{II}'$  and  $G_{II}''$  < 5%.

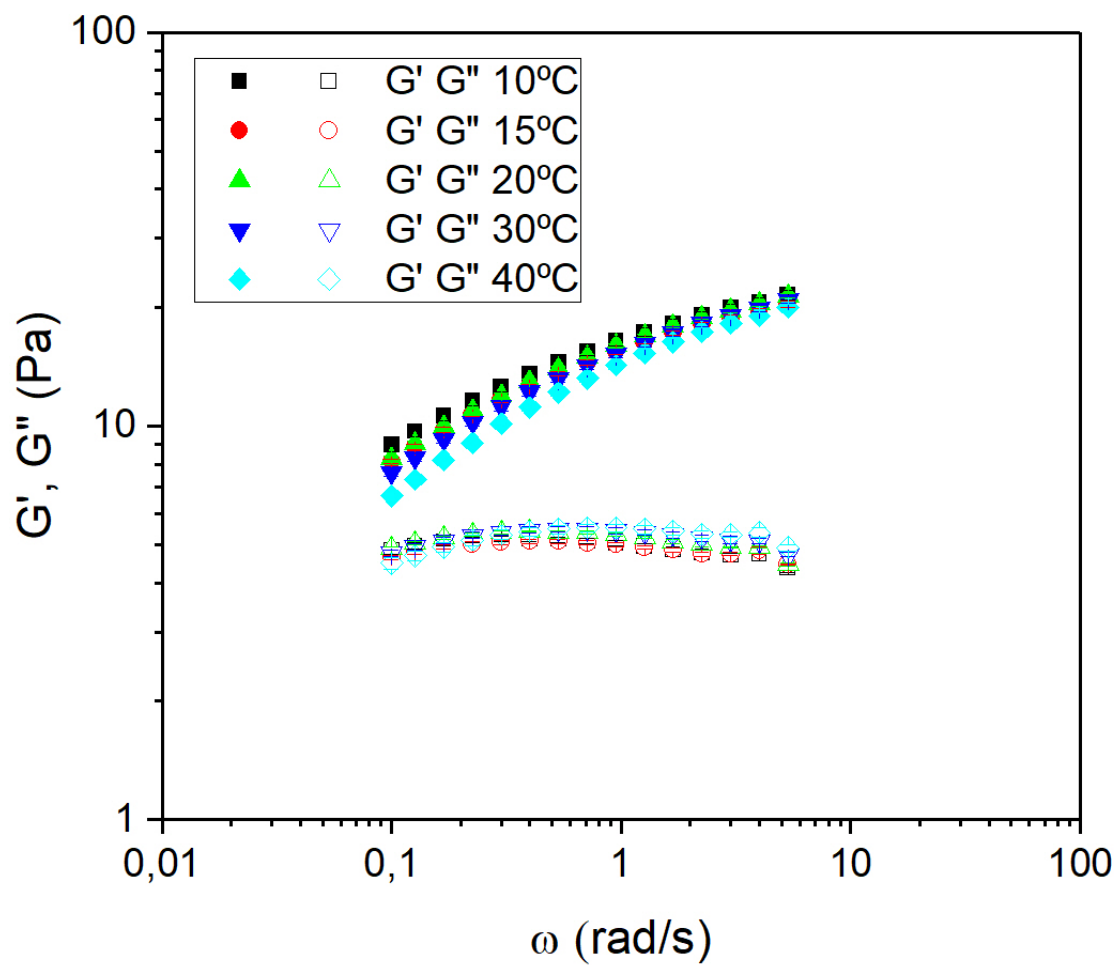
407

408 **Figure 8.** Terminal relaxation times for a 0.5 % (m/m) diutan gum solution as a  
409 function of parallel shear stress and temperature in the (10–40) °C range.  
410 Standard deviation for terminal relaxation times < 5%.

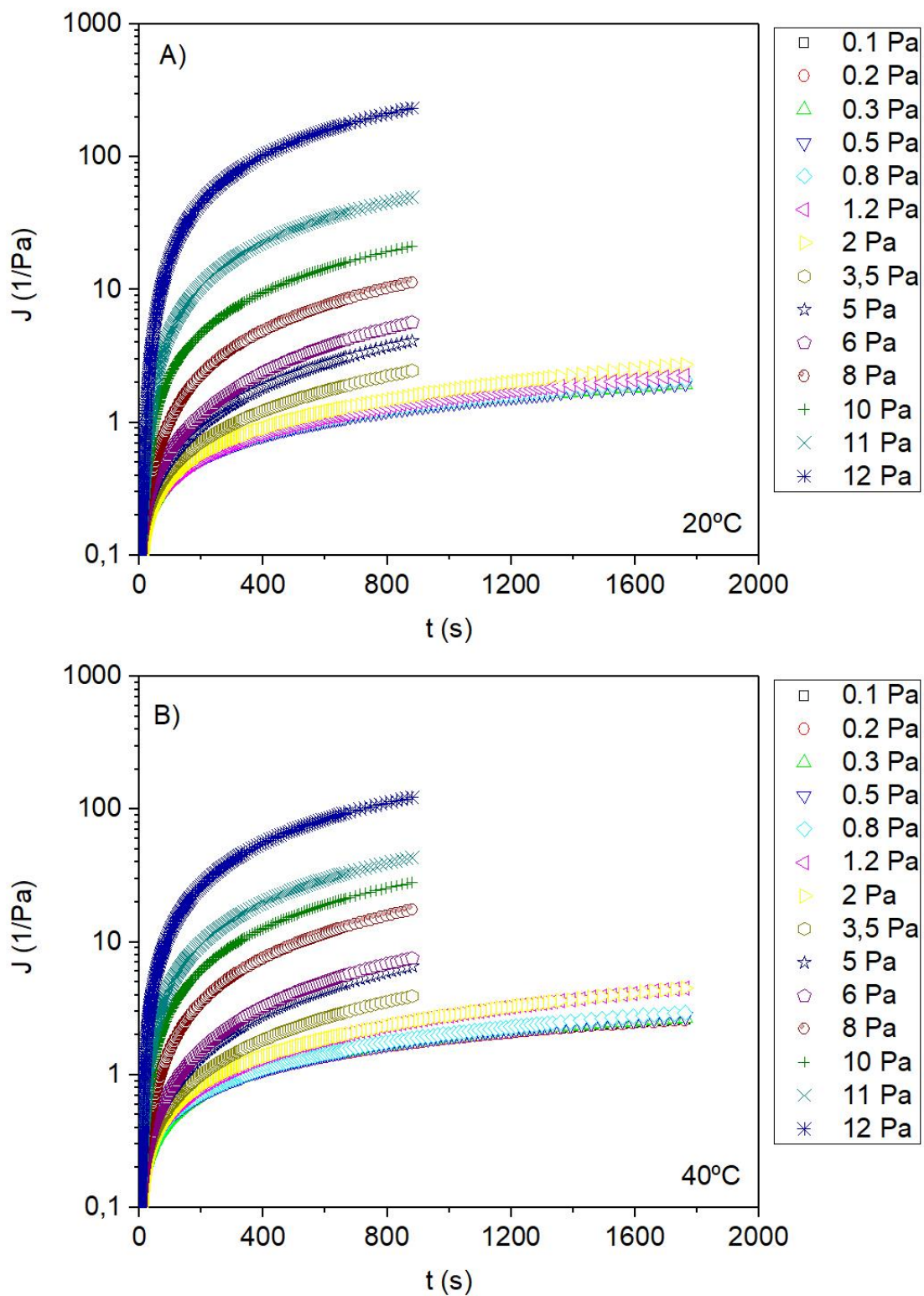
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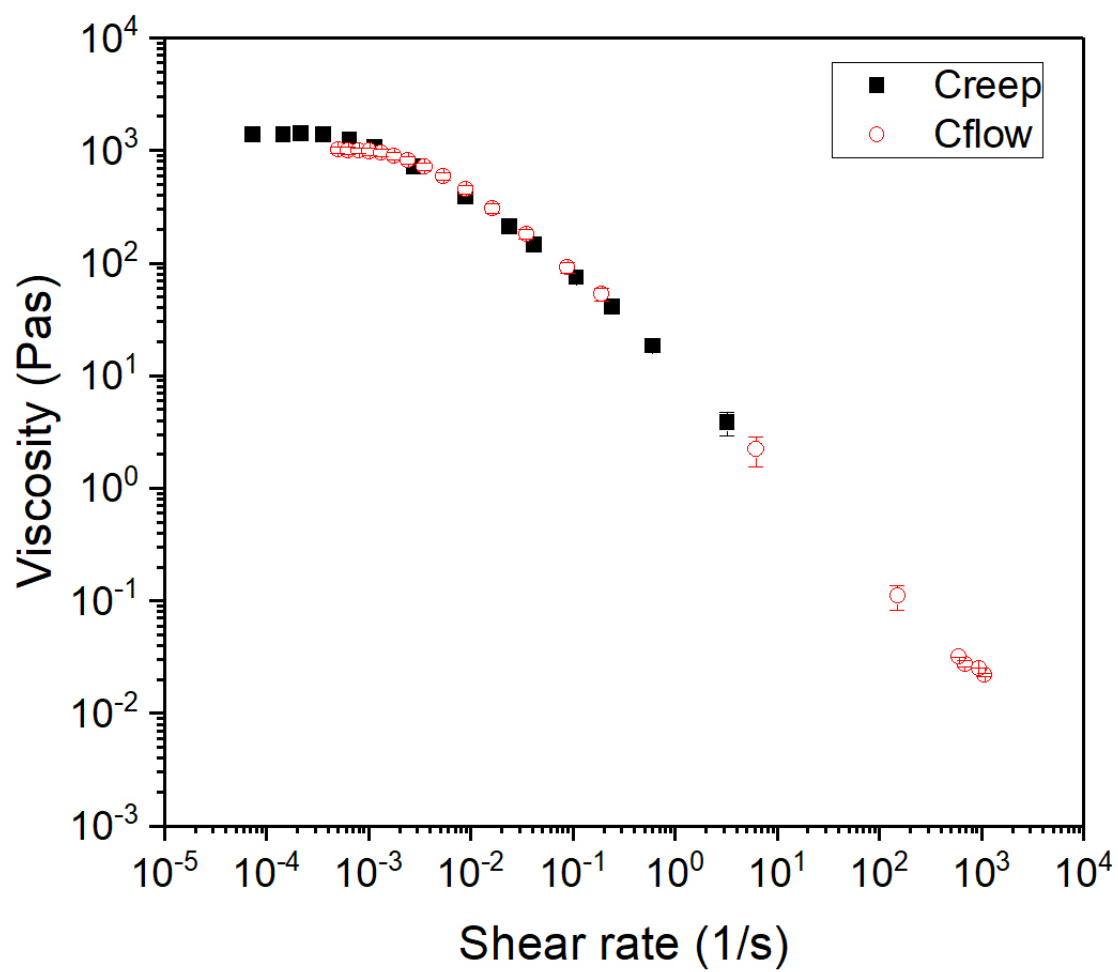


**Figure 1.** Mechanical spectra for 0.5 wt % diutan gum solution as a function of temperature in the (10–40) °C range. Standard deviation for  $G'$ ,  $G''$  < 5%.

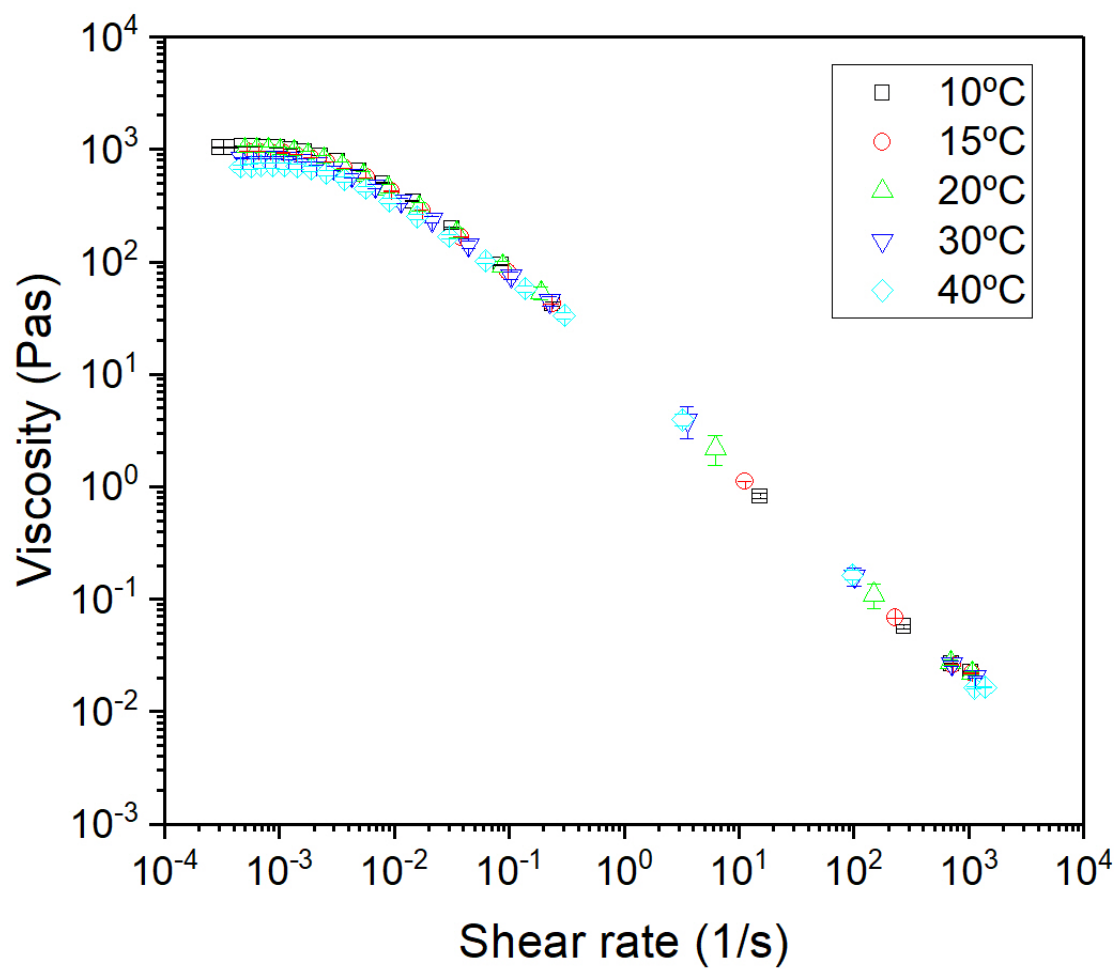


**Figure 2.** Compliance as a function of time for various shear stress. Results obtained by creep compliance tests for a 0.5 wt. % diutan gum solution at 20°C and 40°C. Standard deviation for  $J < 10\%$ .

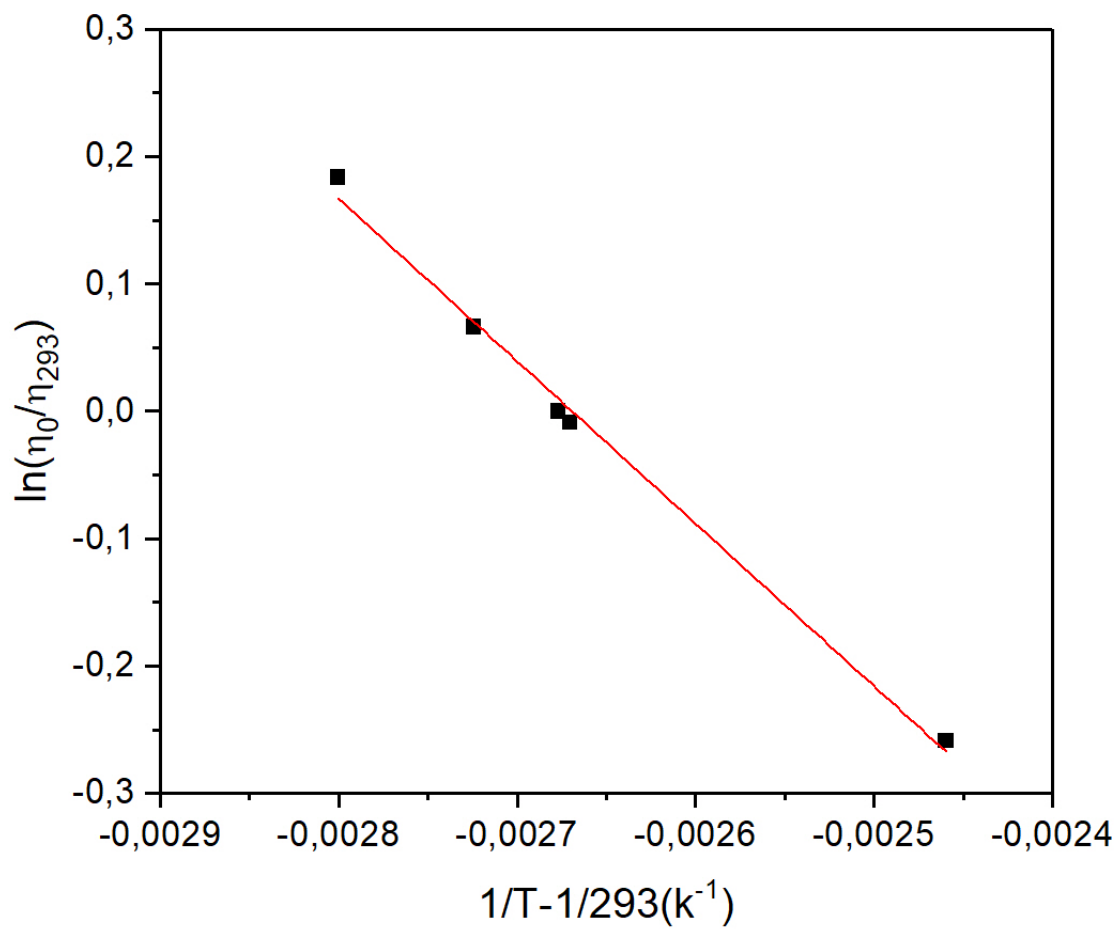




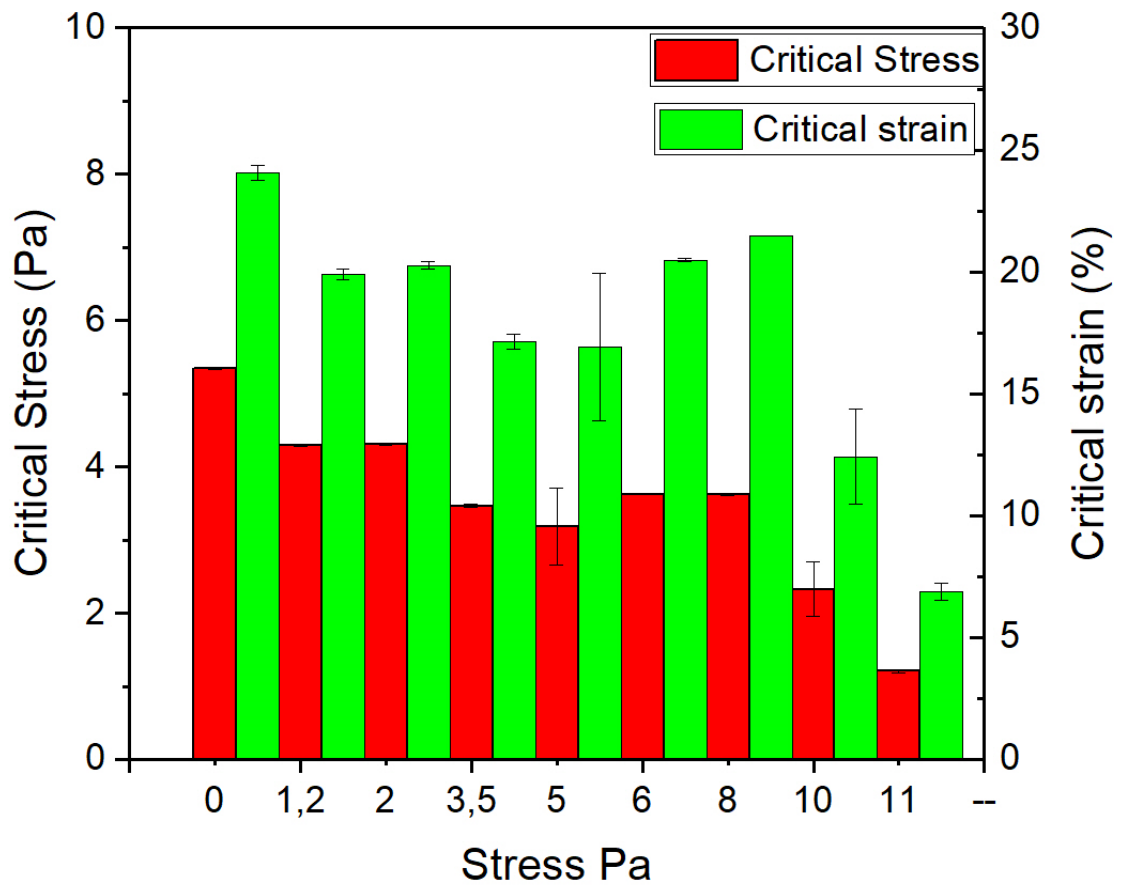
**Figure 3.** Shear rate dependence of steady state viscosity obtained by combining creep compliance tests (closed symbols) and multistep flow curves (open symbol) for 0.5 wt % diutan gum solution at 20°C. Standard deviation for  $\eta < 10\%$ .



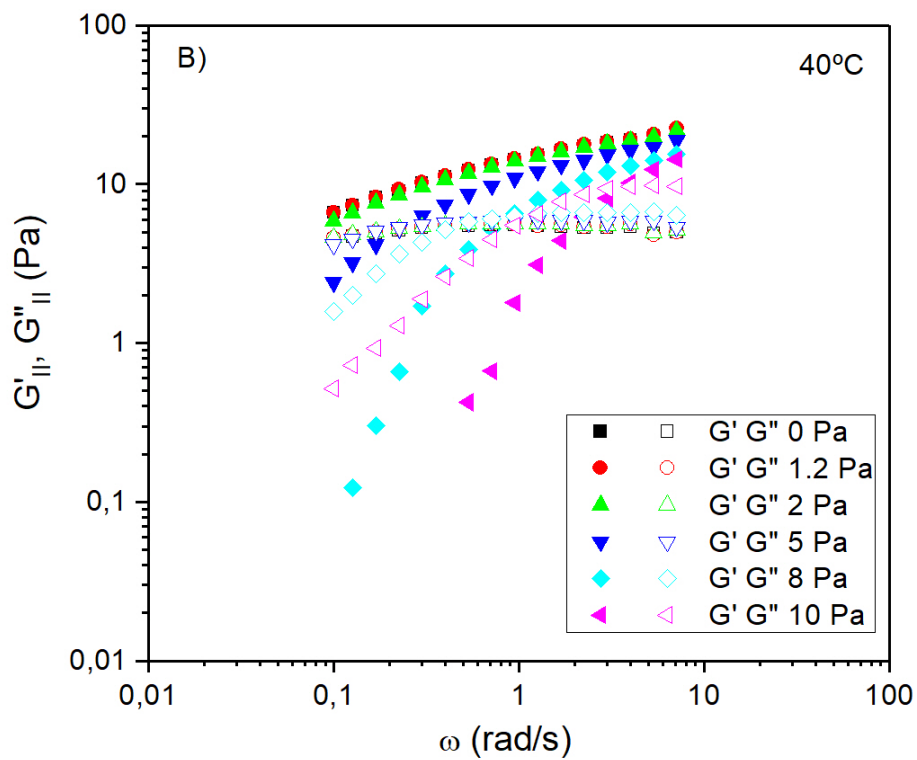
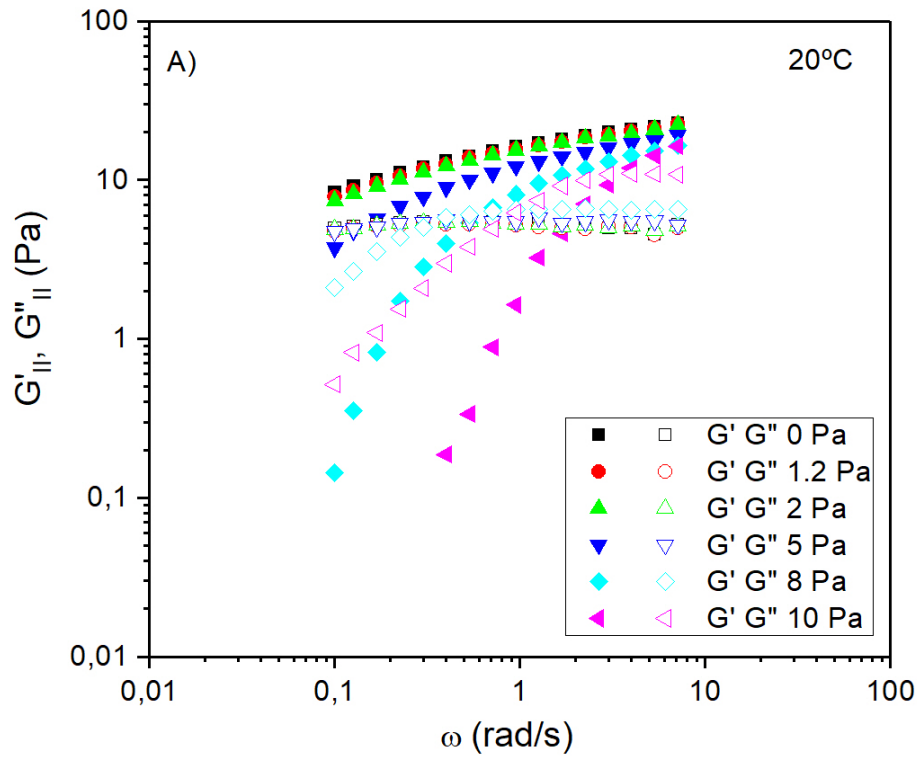
**Figure 4.** Steady shear flow curves for 0.5 wt % diutan gum solution as a function of temperature in the (10–40) °C range. Standard deviation for  $\eta < 10\%$ .



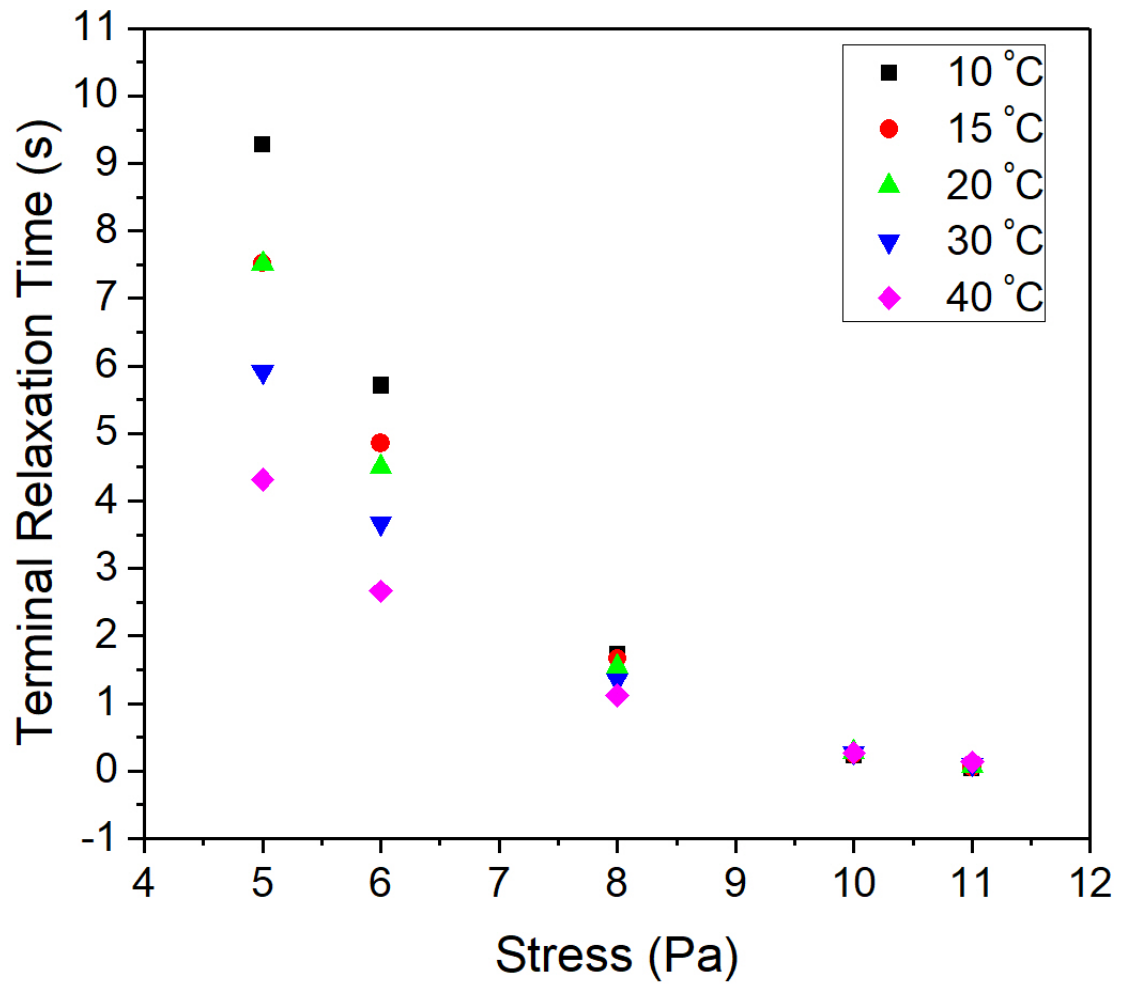
**Figure 5.** Arrhenius-type plot for the temperature dependence of the zero-shear viscosity for 0.5 wt % diutan gum solution. The line shows data fitting to the Andrade equation.



**Figure 6.** Critical stress and strain values for a 0.5 % (m/m) diutan gum solution as a function of parallel shear stress at 20°C. Error bars correspond to standard deviation from the one-way analysis of variance (ANOVA) at a 5% significance level.



**Figure 7.** Parallel storage moduli  $G_{II}'$  and parallel loss moduli  $G_{II}''$  as a function of angular frequency for various parallel shear stress values. For a 0.5 % (m/m) diutan gum solution at 20°C and 40°C. Standard deviation for  $G_{II}'$  and  $G_{II}''$  < 5%.



**Figure 8.** Terminal relaxation times for a 0.5 % (m/m) diutan gum solution as a function of parallel shear stress and temperature in the (10–40) °C range. Standard deviation for terminal relaxation times < 5%.