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ORIGINAL PAPER

Bio-Based Guar-Borate Hydrogels: Processing Effects and Rheological Insights for High-Temperature Applications

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Abstract
The increasing emphasis on environmentally sustainable practices in industry has intensified interest in natural biopolymers for use in advanced material applications. Hydrogel systems composed of natural polymers, such as guar, xanthan gum, or carboxymethyl cellulose, crosslinked with borate or transition metal complexes are particularly relevant due to their renewability and tunable rheological properties. In this study, bio-based guar-borate hydrogels were prepared and rheologically characterised over a temperature range of 25 to 140 °C, using a rheo-reactor apparatus that enables measurements above the solvent's boiling point. Various geometries were employed depending on the viscosity of the formulation, allowing a broad range of shear rates to be explored. Gelation was found to occur rapidly, typically within 10 min; however, inefficient distribution of borate ions in highly viscous guar solutions delayed network formation. The gels exhibited significant increases in elastic properties and shear thickening upon crosslinking. Rheological properties, including gel strength and elasticity, decreased exponentially with temperature. Despite this, the gels retained high viscosity and viscoelasticity up to 100 °C, beyond which a discontinuous gel phase dominated the response. These findings contribute to the understanding of structure–property–processing relationships in natural polymer systems and highlight the potential of guar–borate gels for sustainable, high-temperature applications.

Keywords Guar-borate gel · High temperature · Processing · Rheology

Introduction

Guar gum (GG) is a high-molecular weight, water-soluble polysaccharide, produced from the processing of seeds of *Cyamopsis tetragonoloba*, which has long been used in various fields, including drug delivery [1], food processing [2] and oil and gas recovery [3, 4] due to the ease of modifying its rheological properties. The increasing focus on environmentally friendly practices in the oil and gas industry has increased the interest in natural polymers like GG, which can provide effective solutions without the adverse environmental impacts associated with synthetic additives [5]. Nevertheless, some limitations have been detected in the temperature ceiling of application due to the thermal resistance of polysaccharide biopolymers [6–9].

In hydraulic fracturing operations, an aqueous gel is used to suspend proppant particles, which are used to keep the fracture open once it has been created. The high pressure applied fractures the rock and causes the gel with proppant to flow into the fracture and to propagate the fracture away from the wellbore into the reservoir rock. The gel is then degraded and removed from the proppant-laden fracture, leaving behind open a more permeable pathway for oil and gas to flow from the reservoir rock into the well [3, 4]. Consequently, it is very important that the gel retains a viscosity sufficiently high to generate and propagate the fracture during the high-pressure pumping operations. It must also suspend the proppant particles and prevent them from settling before and during penetration into the fractured rock formation [10].

Crosslinking agents, such as borax (sodium borate), are frequently added to GG to enhance the viscosity of the fluid,

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while keeping the concentration of biopolymer at a minimum [11]. To further reduce concentration, but maintaining the overlap concentration required, extended crosslinkers may be considered [12, 13]. In this context, zirconium chelates, boric acid, boronic compounds, and nanosilica have been incorporated into GG solutions to form hydrogels. The crosslinking reaction of GG occurs between the cis-hydroxyl groups of galactose units and the hydroxyl ions of the crosslinker. The concentration of borate ions in solution depends on the pH, as borate exists in equilibrium with boric acid (pKa = 9.0–9.2). A minimum borate concentration and a pH above 9 are necessary to develop 2:1 complexes, resulting in a physical three-dimensional structure and an increase in viscosity and elasticity. Details of the reaction kinetics have been reported elsewhere [14, 15]. Complexation with transition metal chelates can occur via irreversible covalent bonds within the pH range of 2–11. Zirconium chelates can agglomerate to form polyvalent colloidal species that enhance the performance of the borate crosslinker [16–19]. Functionalised polyols and nanoparticles have emerged as a new generation of crosslinkers [20, 21].

The rheology of GG gels depends on several factors such as GG and crosslinker concentration, side substitution groups in the biopolymer chain, pH, temperature and pressure [22]. The study of these variables is essential to develop suitable products for optimal application in each field [23]. It is known that the concentration of borate ions in solution depends on pH, temperature and concentration of borax [24], consequently, the rheology of guar crosslinked with borate (GB) gels depends significantly on these variables as it has been reported in early studies by Pezron and co-workers [25, 26]. For the oil industry, suitable products can be obtained above a critical overlap concentration, situated at the transition from the dilute to the semi-dilute regime [27].

Pezron et al. [28] studied the effects of both polymer and borate concentrations, ionic strength, pH, and temperature on the viscoelastic properties of semi-dilute galactomannan solutions, concluding that the rheology of these gels can be characterised by the elastic plateau modulus and a characteristic frequency at the maximum of loss modulus. The evolution of the elastic plateau modulus varies linearly with borate concentration and potentially with the concentration of biopolymer. The characteristic frequency was related to the complexation kinetics and pH. The time–temperature superposition was applied using vertical shift factors due to the exothermic nature of the complexation reactions. Kesavan and Prud'homme [29] studied the gelation of hydroxypropyl guar (HPG) and GG by borate ions ($B(OH)_4^-$) as function of both temperature (10-65ºC) and pH (6.36–9.5). These authors separated the contribution to stress due to chain entanglements and chain association, concluding that the GB gel obeys both time–temperature and time–pH superposition using vertical and horizontal shift factors. The temperature superposition reduced moduli fitted to a single Maxwell relaxation time, indicating the simplicity of the rheological response. Wang et al. [30] investigated the effect of pH on the rheology of 0.50 wt% solution crosslinked with borate in the range of temperature from ambient to 80ºC. These authors concluded that maximum viscosity was obtained in the range of pH from 8.5 and 12, decreasing for higher pH values, according to previous studies reported in the literature for systems of similar composition. In this pH range was obtained the highest values of elasticity, gel strength and thixotropy.

Sun and Boluk [31] studied the impact of cellulose nanofiber (CNF) on the shear rate-dependent viscosity, viscoelasticity and the proppant suspension capability of a guar fracturing fluid solution formulated with 0.25% of GG and crosslinked with borate. The presence of CNF significantly influenced the rheological behaviour of GB gels, including the apparent viscosity and viscoelasticity. CNF concentration increased the viscosity at low shear rates and decreases it at high shear rates. The elasticity measured by the value of the elastic modulus of the gels was significantly improved with a scaling exponential factor of 2.8. Consequently, the static and dynamic proppant suspending capacities were improved. On the other hand, it has been demonstrated that GG gels crosslinked with nanosilica exhibit higher elastic (G') and viscous (G") moduli compared to GB gels [32]. Modified GG derivatives, such as HPG and carboxymethyl guar (CMG) may enhance both the thickening ability of guar gum [33] and its thermal stability to degradation [5, 34]. The rheology and reaction kinetics of guar and CMG derivatives crosslinked with boric acid and zirconium chelate have been compared. The zirconium crosslinker produced stronger gels at a given crosslinker concentration, but CMG resulted in less cross-linking than GG due to structural impediments [35]. The structure and properties of the GB gels compared with those of Scleroglucan/borate have been characterised using AFM [36].

The trends in the preparation of GG products have focused on enhancing the rheological performance and resistance in extreme conditions such as high temperature, pressure and high salinity environments [16, 34, 37] to cover oil and gas operations in deeper scenarios[6]. In this context, less attention has been devoted to the rheological behaviour of these gels in the region of high temperature above the boiling point of the solvent [4]. To further investigate the role that temperature plays in the rheological behaviour of guar gels, this paper explores the processing and rheological properties of GB gels over a wide range of temperatures, extending the study to high-temperature regions above the boiling point of the solvent by employing pressurised geometries to prevent water vaporisation. The results could be applied

to the optimisation of oil and gas recovery operations at elevated temperatures.

Materials and Methods

Materials

Commercial food-grade GG with $M_w = 2 \times 10^6$ g/mol (Sigma Aldrich) was used without further purification. Sodium tetraborate decahydrate (borax), supplied by Sigma Aldrich (Germany), was used as crosslinker.

Sample Preparation

Two concentrations of GG solution, 0.25 and 0.50 wt%, were prepared by adding GG powder (without purification) to deionised water. A 0.05% wt% sodium azide was added to the solution as a preservative. These solutions were gently stirred using a magnetic stirrer for 15 min at ambient temperature. The samples were maintained at rest to fully hydrate for 24h. Subsequently, high shear homogenisation was carried out for 5 min to disperse any lumps. This duration was sufficient to achieve complete homogenisation while minimising the risk of mechanical degradation of the polymer.

Crosslinked borate gels (GB) were formulated with guar-borate mass ratios of 2:1 and 8:1. All the fracturing gels were prepared by dispersing the required amount of borax powder in the guar solution using a four-blade impeller at 250 rpm at 60ºC for a mixing time of 15 min, previously optimised in the processing study. After mixing, the pH was measured and adjusted if necessary to pH 9 by adding a 0.1 M NaOH solution. Later, the mixtures were maintained in an oven for 15 min at 60ºC and subsequently cooled to room temperature to produce a homogeneous gel. Again, the pH was checked and, if necessary, readjusted to pH 9.

The kinetics of the mixing process were studied in a closed rheo-reactor (Fig. 1). This rheo-reactor consists of a cylindrical vessel pressure cell D400 (36-mm diameter, 60-mm height), equipped with a four-blade geometry (25-mm diameter) that was magnetically coupled with the motor of a controlled-stress MCR301 rheometer (Anton Paar, Austria). This instrument permits the monitoring of the evolution of torque over time and has been successfully employed in several rheo-mixing studies [38, 39]. Approximately 60 mL of guar solution previously prepared were introduced into the rheo-reactor and mixed at 250 rpm for 10 min to achieve thermal homogenisation. Subsequently, the borax

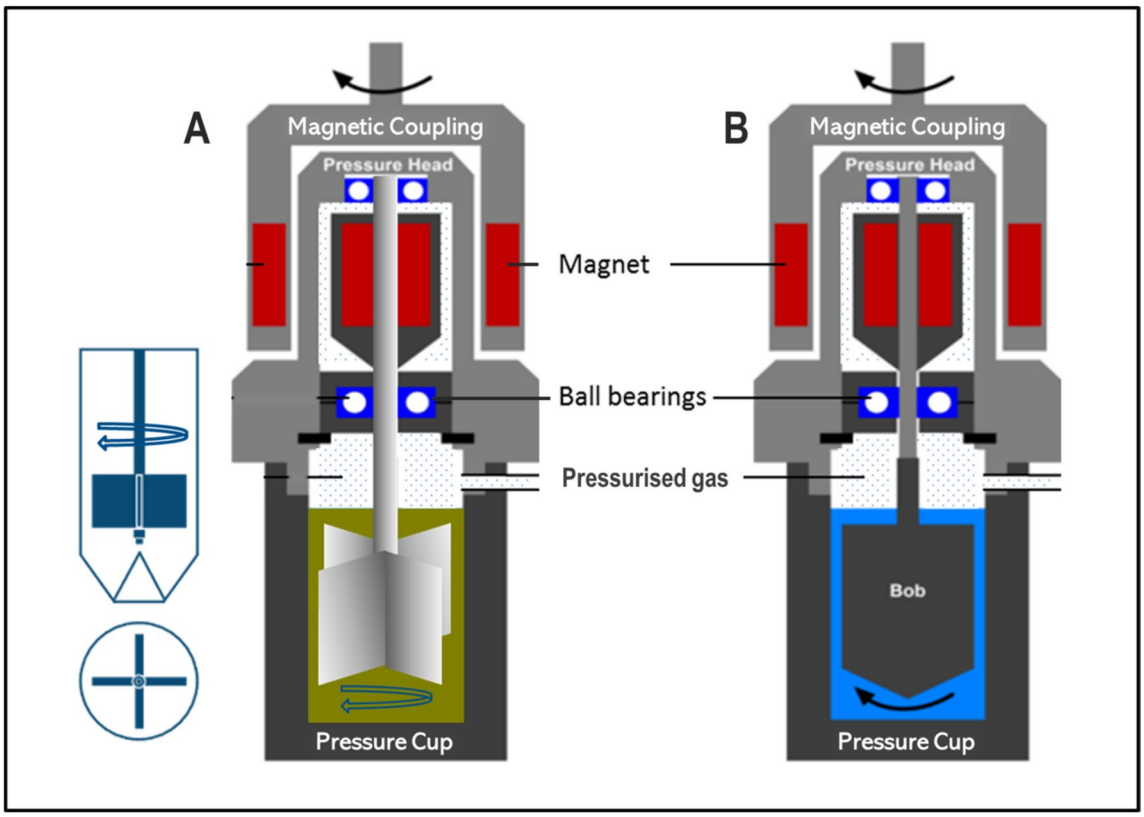

Fig. 1 D400 pressure cell used in the study. (**A**) Configured as a rheo-reactor with a non-conventional four-blade geometry. (**B**) Employed with pressurised geometries to perform steady-state viscosity measurements in the 80–160 °C range

powder was meticulously incorporated into the guar solution, which was then blended by stirring at 250 rpm and monitored for a period of 30 min. All samples were processed at a temperature of 60°C. Following processing, a series of rheological tests were conducted using the four-blade geometry.

Rheological Measurements

The rheological characterization was carried out using two controlled-stress rheometers, a Physica MCR-301 and a Haake MARS II (Thermohaake, Germany) both equipped with the most appropriate geometry such as coaxial cylinders (CC27, CC27PR and Z41), double gap and coaxial cylinder pressurized geometries (DG35, CC30PR).

Frequency sweeps, from 0.01–100 rad/s, were then performed in the range of 25-80ºC, using conventional geometries and pressurized geometries at higher temperatures, selecting a shear stress value within the linear viscoelasticity range, which was previously determined at each temperature by stress sweep tests at the frequency of 1 rad/s.

Steady-state viscosity curves were recorded at 25, 40, 60, and 80 °C using conventional coaxial cylinder geometries (CC27 and CC27PR) open to the atmosphere. At higher temperatures (80–160 °C), the D400 pressure cell was fitted with pressurised coaxial cylinder geometries (DG35 and CC30PR), employing 50 bar of inert gas (N_2) to prevent water boiling. Samples were loaded at ambient temperature, and the measurement temperature was stabilised for one hour. A solvent trap was used to minimise water loss during tests conducted with conventional geometries.

Results and Discussion

Processing Rheokinetics of Guar-Borate Gels

The processing of GB gels yields fluids with complex rheological behaviour, where laminar and extensional flows interact concurrently with distributive mixing. The initial contact stage, coupled with transport phenomena, mixing conditions, and the medium's intrinsic rheological complexity, can markedly influence reaction kinetics, thereby affecting the properties and overall quality of the final product. In this context, rheo-reactor devices prove to be valuable tools for examining and monitoring changes in rheological behaviour both during and after processing [38, 40]

The evolution of viscosity during the processing of GB gels is shown in Fig. 2A. It can be seen that there is a first region (from 0–10 min) characterised by an initial slight decrease in viscosity which gradually trends towards a final constant value. This decrease in viscosity was attributed to the stabilisation of the processing temperature in the bulk solution. After the addition of the borate salt, at a processing time of 10 min, there is a sharp increase in the viscosity of the solution due to the guar-borate crosslinking reaction [25, 26]. The characteristic slope, which is associated with the kinetics of the crosslinking reaction, along with the maximum viscosity attained in the processing curves, depends on the GB concentration. The systems with a GB ratio of 8:1 show both a lower slope and a lower maximum viscosity than the systems with a GB ratio of 2:1. This is in agreement with the previously published literature, which states that the GB crosslinking density is proportional to the borate content [29]. As the borate concentration increases, there is a significant increase in viscosity due to the increased crosslinking density for a given GG solution. In addition, the processing time required to reach maximum viscosity decreases slightly with borax content (from 105 to 94 s). After the maximum viscosity, the processing time has different effects on the structure depending on the GB concentration ratio. For a lower borate concentration (8:1 ratio), a clear shear destruction (30% viscosity loss) of the initial crosslinked structure is observed with processing time, with a tendency to reach a constant viscosity value at longer processing times. Conversely, for a higher borate concentration (GB ratio of 2:1), the viscosity remains at the maximum value over the processing time, indicating the formation of a stronger gel.

In the more concentrated solution (0.50 wt% GG), both the slope and the maximum viscosity values appear unaffected by the GB ratio, which is attributed to the comparable viscosities exhibited by these gels under the given processing conditions. In both cases, a minimum reaction time of 10 min is required to reach the maximum viscosity, longer than that required for GG 0.25 wt%. The overshoot of the viscosity is also more pronounced for GG 0.5 wt% GB ratio of 8:1, with a tendency to recover the values of the maximum along the processing time. However, for GG 0.25 wt%, a slight loss in viscosity is observed, which is more pronounced for the GB ratio of 8:1. More scatter in the viscosity is observed for stronger gels, which is attributed to heterogeneity of the system due to the overlap of macromixing and gel breaking-reconstitution processes.

The mixing time plays an important role in the kinetics of the gelation process for concentrated systems, such as GG 0.50 wt% and GB ratio of 2:1. Figure 2B compares the viscosity evolution with time for two batches of the same concentration (GG 0.50 wt% GB ratio of 2:1) where the borate is added quickly (within 30 s) and slowly (over 300 s). The mixing process strongly influences the kinetics of gel formation, an initial mixing region is followed by a reaction region, as can be seen from the process curve in Fig. 2B. In the case of rapid addition, the mixing time is approximately

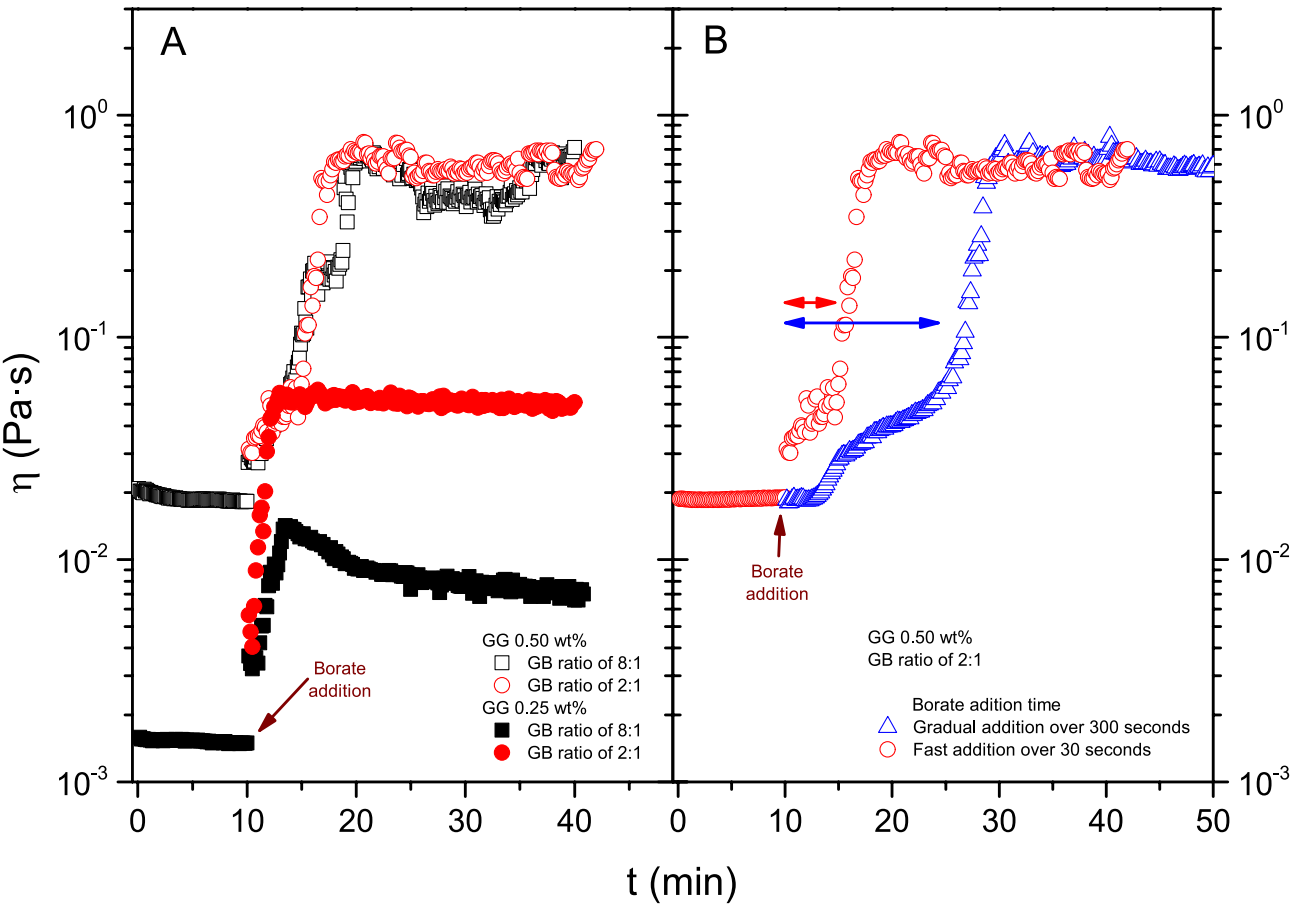

Fig. 2 Evolution of viscosity over time during the processing of GB gels in the rheo-reactor at 250 rpm (approximately 9 s^{-1}) and 60 °C. (**A**) Effect of borate content and GB ratio on viscosity. (**B**) Effect of borate addition time on viscosity

5 min. During this time the viscosity of the system slowly increases to $5 \cdot 10^{-2}$ Pa·s. Shortly afterward, the viscosity suddenly increases up to the maximum value in a reaction time of 5 min. In the batch processed with slow addition, there is a mixing dead time of approximately 3 min. The viscosity increases up to $3 \cdot 10^{-2}$ Pa·s during the addition time, the mixing time is approximately 10 min, indicating a slower mass transfer kinetics than in the case of rapid addition. As expected, the reaction time is also near to 5 min for the batch with slow addition. Finally, the viscosity values reached after the mixing and reaction time is practically the same for both batches.

After the processing time, the evolution of the storage and loss moduli was measured with the rheo-reactor as a function of time, at a temperature of 60ºC and a frequency of 6.28 rad/s, applying a low deformation to ensure that the gel remains in the linear range. No significant evolution of the moduli and loss tangent was observed during the rest time of 1 h (results not shown). It can be concluded that the complexation reaction was completed during the processing of time 10 min, rapid addition, 20 min slow addition, under these mixing conditions.

The rheo-reactor was cooled to room temperature, 25ºC, for 3.5 h and stored for 24 h, followed by a temperature sweep at a heating rate of 1ºC/min, pressurizing the system with 50 bars of N_2 to avoid vaporization of the solvent. The evolution of the moduli versus temperature is presented in Fig. 3.

Figure 3 shows that the rheo-reactor used is a suitable device for measuring the evolution of the viscoelastic properties as a function of time and temperature [38]. As the temperature increases, the storage modulus decreases progressively, while the loss modulus either remains constant or increases, until both moduli converge at the crossover point. This behaviour can be attributed to a reduction in crosslinking density with rising temperature, which affects the elastic modulus (G′) more significantly than the viscous modulus (G”). An increase in temperature decreases the crosslinking density, which affects the elastic properties more than the viscous ones. At higher temperatures, a significant decrease in the values of the moduli is observed. The crossover point occurs at temperatures of 82 °C and 70 °C for gels formulated with GG 0.50 wt% solution and GB ratios of 2:1 and 8:1, respectively. However, the rheo-reactor equipped with

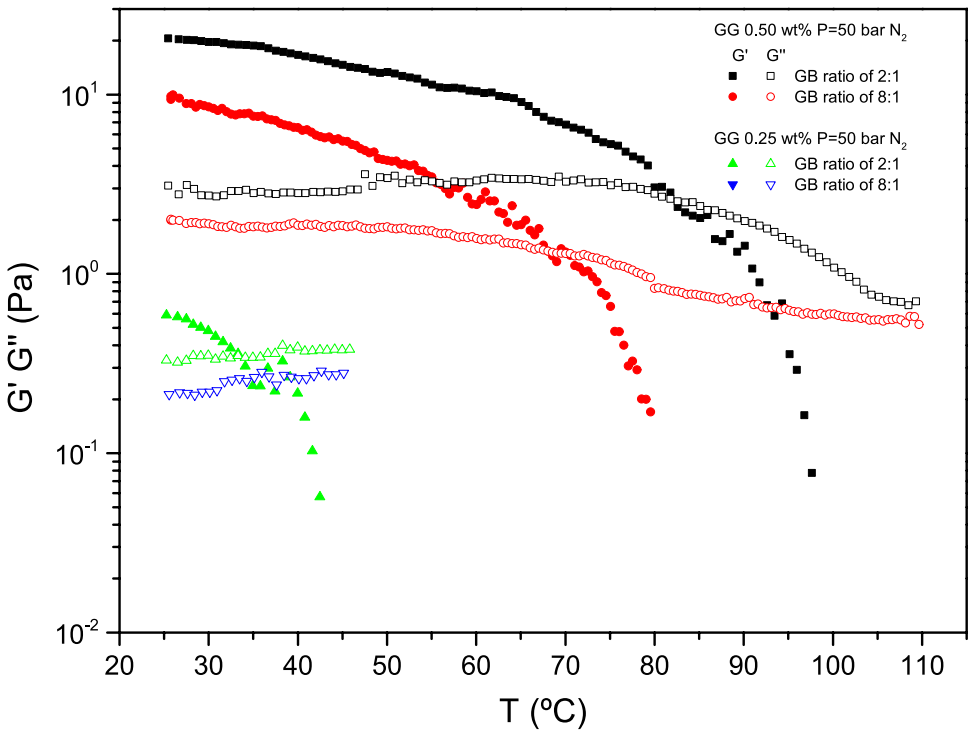

Fig. 3 Oscillatory temperature sweeps of the GB gels after processing, measured at a frequency of 6.28 rad/s using the rheo-reactor equipped with a four-blade geometry

a four-blade geometry does not have the necessary sensitivity to measure the elastic behaviour of the gels with temperature when they were prepared with a GG concentration of 0.25 wt%, resulting in an inaccurately determination of the crossover point. These results highlight the weakening of GB gels with temperature as reported by several authors [4, 6, 28, 34], which is much more accelerated in the high temperature region, near the crossover point. These gels may reduce their ability to suspend proppant particles and keep the fracture open at high temperatures due to weakening of the gel structure, as evidenced by a decrease in storage modulus (G') and crossover point at relatively low temperatures (70-82ºC), which is dependent on borate concentration.

Effect of Temperature on the Dynamic Behaviour of Gels

Figure 4A shows the evolution of both storage (G') and loss (G'') moduli versus frequency, in the range of temperatures from 20 to 120ºC, for the gels prepared with GG 0.50 wt% at a GB ratio of 2:1. As can be observed, at temperatures below 80ºC, the G' is higher than the G'' in the region of medium to high frequency, revealing that a gel is developed in the solution. This behaviour is in agreement with that previously reported in the literature for GB gels of similar concentrations [28, 29].

At temperatures above 80ºC, the region in which the solution shows gel behaviour is strongly reduced, G' shows higher values than G'' only at higher frequencies. Thus, at 120ºC, the solution does not behave as a viscoelastic gel and the flow terminal region is clearly evidenced over almost all the range of frequencies tested. When the gel is prepared with a lower borate concentration, GB ratio of 8:1, a well-developed gel is also observed at temperatures below 60ºC. Lower values of the moduli are observed for lower borate concentration and a lower temperature resistance of the gel can be deduced from Fig. 4B. For a GB ratio of 8:1, the terminal flow region covers the whole range of frequency tested at temperatures above 80ºC.

For a less concentrate GG solution (0.25 wt%), near the limiting value to form viscoelastic gels when GG is crosslinked with borate [27], a well-developed gel is also observed if the borate concentration is relatively high at temperatures below 60ºC, as can be observed in Fig. 5A. At a given temperature, the gel formulated with a less concentrated GG solution (0.25 wt%) show weaker rheological properties than that formulated with the more concentrated solution (0.50 wt%); however, according to Lei and Clark [27], the limit of GG 0.25 wt% can be considered as a threshold GG concentration to form gel for engineering proposes. In addition, the gel formulated with less GG content is more sensitive to temperature, mainly at high GB ratio. As can be seen in Fig. 5B, the gel with GB gel ratio of 8:1 shows predominant elastic behaviour only at the temperature of 25ºC. It is also interesting to note, comparing Figs. 4 and 5, that the gel formulated with GG solution of 0.25 wt% shows a terminal region with G' and G'' slopes of 2 and 1 in log scale, respectively, whereas gels formulated GG 0.50

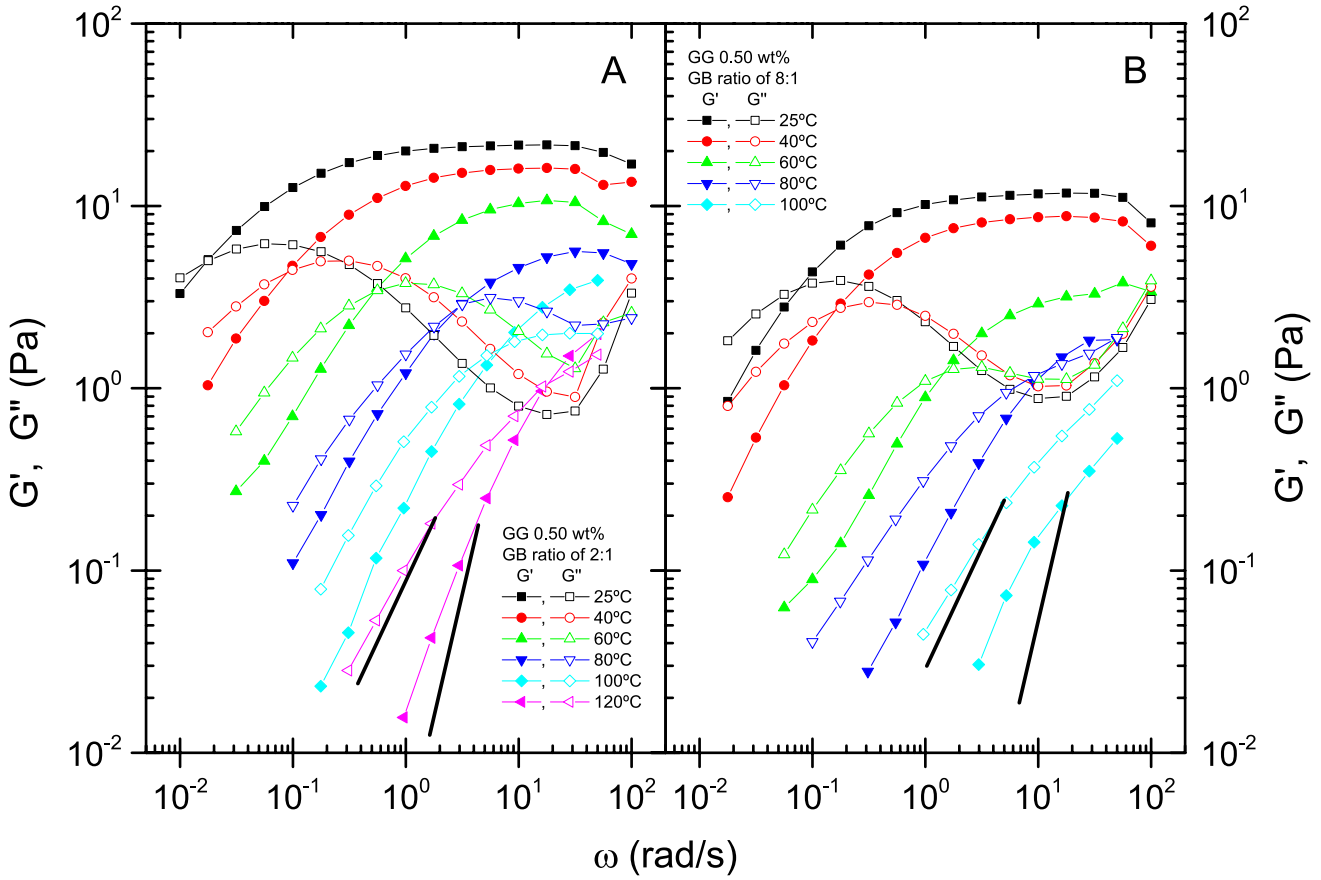

Fig. 4 Dynamic behaviour as a function of temperature for 0.50 wt% GG gels with different GB ratios. (**A**) GB ratio of 2:1. (**B**) GB ratio of 8:1. The black lines indicate reference slopes of 1 and 2 on a logarithmic scale

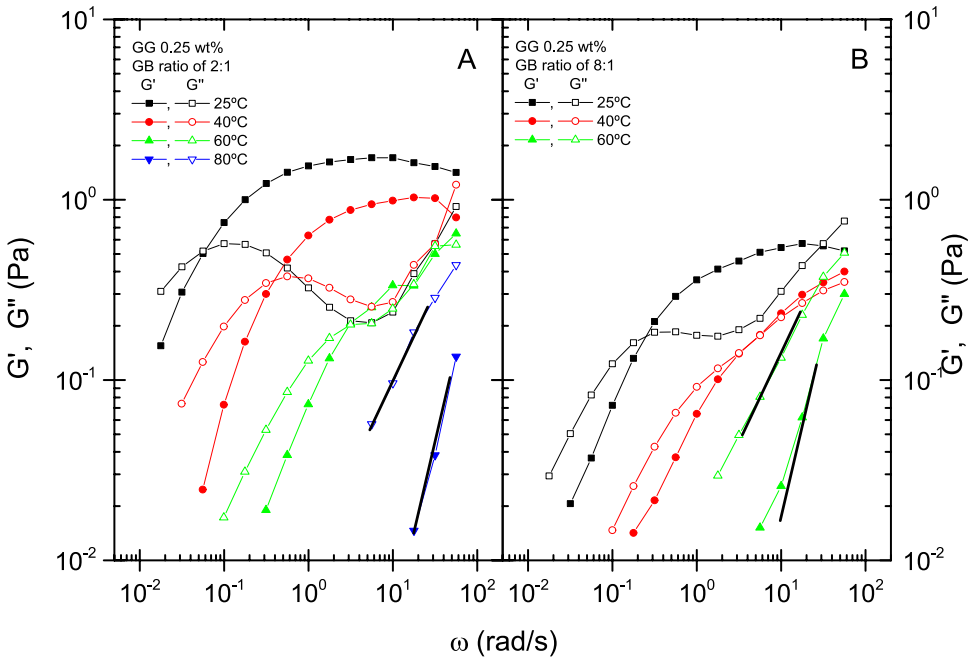

Fig. 5 Dynamic behaviour as a function of temperature for 0.25 wt% GG gels with different GB ratios. (**A**) GB ratio of 2:1. (**B**) GB ratio of 8:1. The black lines indicate reference slopes of 1 and 2 on a logarithmic scale

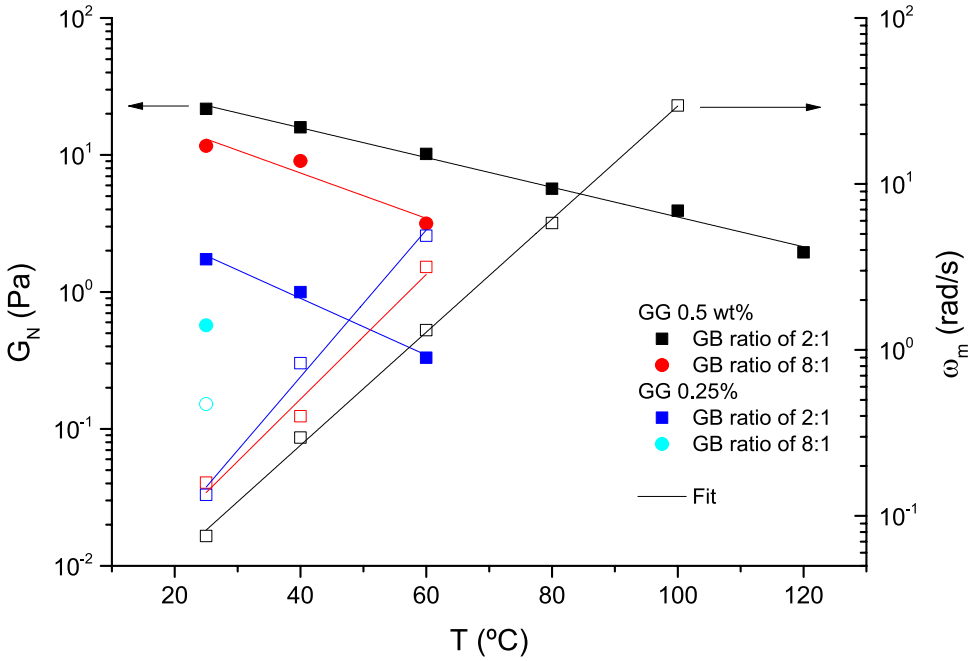

Fig. 6 Plateau modulus (G_N): value of the elastic modulus measured at the frequency corresponding to the maximum of the loss modulus (solid symbols), and characteristic frequency (ω_m): frequency at the maximum of the loss modulus (open symbols), as a function of temperature for the GB gels studied

Table 1 GB gel crossover point parameters as a function of temperature

Temperature (º C)	GG 0.50 wt% GB ratio of 2:1		GG 0.50 wt% GB ratio of 8:1	
	ω_{Cross} (rad/s)	G_{Cross} (Pa)	ω_{Cross} (rad/s)	G_{Cross} (Pa)
25	0.0177	4.910	0.0795	3.610
40	0.0905	4.300	0.1620	2.630
60	0.5630	3.410	1.4400	1.210
80	2.4200	2.520	9.3900	0.675
100	6.9900	1.640		
120	17.9000	1.000		
	GG 0.25 wt% GB ratio of 2:1		GG 0.25 wt% GB ratio of 8:1	
25	0.0574	0.519	0.2570	0.176
40	0.3930	0.352	4.0600	0.155
60	2.8900	0.200		

wt%, reach the terminal flow region with slopes less than 2 and 1. This fact would indicate that both temperature and shearing time break the continuity of the network linkages, but a discontinuous gel phase remains at high temperatures due to partial thermal degradation.

Pezron et al. [28] characterised GB gels based on two parameters the plateau modulus (G_N) [41], proportional to the density of crosslinking of the network, that can be considered as a estimation of the gel strength, and a characteristic frequency (ω_m), at the maximum in G'', whose inverse value gives an indication of the longest relaxation time. These parameters are shown in Fig. 6. In addition to the cross-over point parameters are displayed in Table 1 for the gel samples formulated.

As can be observed in Fig. 6, the plateau modulus decreases linearly on a semi-logarithmic scale as temperature increases up to 120 °C, according with Eq. (1):

$$log\,(G_N) = a \cdot T + b \tag{1}$$

with a negative slope, a, of −0.011, for the gel GG 0.50 wt% with a GB ratio of 2:1. This slope decreases in value up to −0.016, as the borate concentration decreases, sample with GB ratio of 8:1. This fact indicate an increase in the rate of destruction of the gel strength that can be explained due to the lower borate concentration available for crosslinking as the temperature increases [25, 26]. When the GG concentration decreases to 0.25 wt%, the negative slope increases to a value of −0.021, for the GB ratio of 2:1, indicating not only a gel with lower strength but also even a more sensitivity of the gel strength to temperature. In addition, the characteristic frequency, ω_m, increases exponentially with the temperature, with a similar model to that of Eq. (1) in semi-log scale. For gels prepared with a GG concentration of 0.50 wt% and a low GB ratio, ω_m increases with a coefficient of 0.034 up to the temperature of 100°C. As the borate concentration decreases or the GB ratio increases, the slope of ω_m increases up to a value of 0.038 for GG 0.25 wt% gel with a 2:1 GB ratio, indicating a shorter frequency range of gel-like behaviour. The reduction of the frequency zone in which the gel-like behaviour occurs when guar and/or borate concentration decreases, probably is due to the lower borate availability. A decrease in GG concentration increases the slope even if the guar-borate ratio is kept at 2:1. The same tendency with temperature and concentration

follows modulus and frequency at the crossover point as can be seen in Table 1. These parameters have been selected as reference values for other authors [28, 29, 36].

According to the reptation model [42, 43], the inverse of the characteristic frequency is a good indication of the longest relaxation time, when the system is represented by only one Maxwell element. The longest relaxation time has been considered the reptation time. Nevertheless, these systems seem to be more complex, and a unique relaxation time may not represent properly the relaxation behaviour. In the case of these systems, reptation is hampered by the complexation of guar-borate. Consequently, the longest relaxation time can be considered as the exchange rate of crosslinking formation [36].

Effect of Temperature on the Viscous Flow Behaviour of Gels

Figure 7 shows the viscosity curves for both 0.50 wt% (Fig. 7A) and 0.25 wt% (Fig. 7B) mass concentration of GG solutions in the range of 25-140ºC, as reference flow curves for samples without any borate added. The viscosity is strongly influenced by the GG concentration. The flow curve for GG 0.50 wt% is characterised by a Newtonian plateau at low shear rates and pronounced shear thinning at high shear rates over the entire temperature range studied. In contrast, the GG 0.25 wt% solution exhibits a slight pseudoplastic behaviour at shear rates higher than 10 s^{-1} between 25 and 80ºC, and Newtonian behaviour at temperatures above 80ºC. This rheological behaviour is typical for galactomannan dispersions as has been previously reported elsewhere [44]. The relatively small non-Newtonian effect displayed by both biopolymer solutions is related to the alignment and deformation of transiently elongated chains by the shear imposed [45]. Furthermore, the effect of concentration on the viscosity values is principally due to the increased entanglement of the polymer chains in solution [46]. Consequently, higher solution viscosities are observed for higher guar concentrations.

In addition, as is expected, the viscosity values observed decrease with increasing temperature over the whole range of shear rates investigated [47]. The reduction of viscosity may originate from changes in the solvency of the medium for the guar molecules and, consequently, results in a less entangled system [48]. At temperatures above 100ºC, the higher levels of energy available could lead to the thermal degradation of the polymer chains in solution [49]. Additionally, it is clear that the Newtonian plateau is extended to higher shear rates with increasing temperature, particularly for 0.25 wt% GG. The increase in the onset of shear-thinning could be related to the formation of weak temperature-dependent intermolecular associations [45, 47].

The viscosity data were correlated using the Williamson's model (Eq. 2):

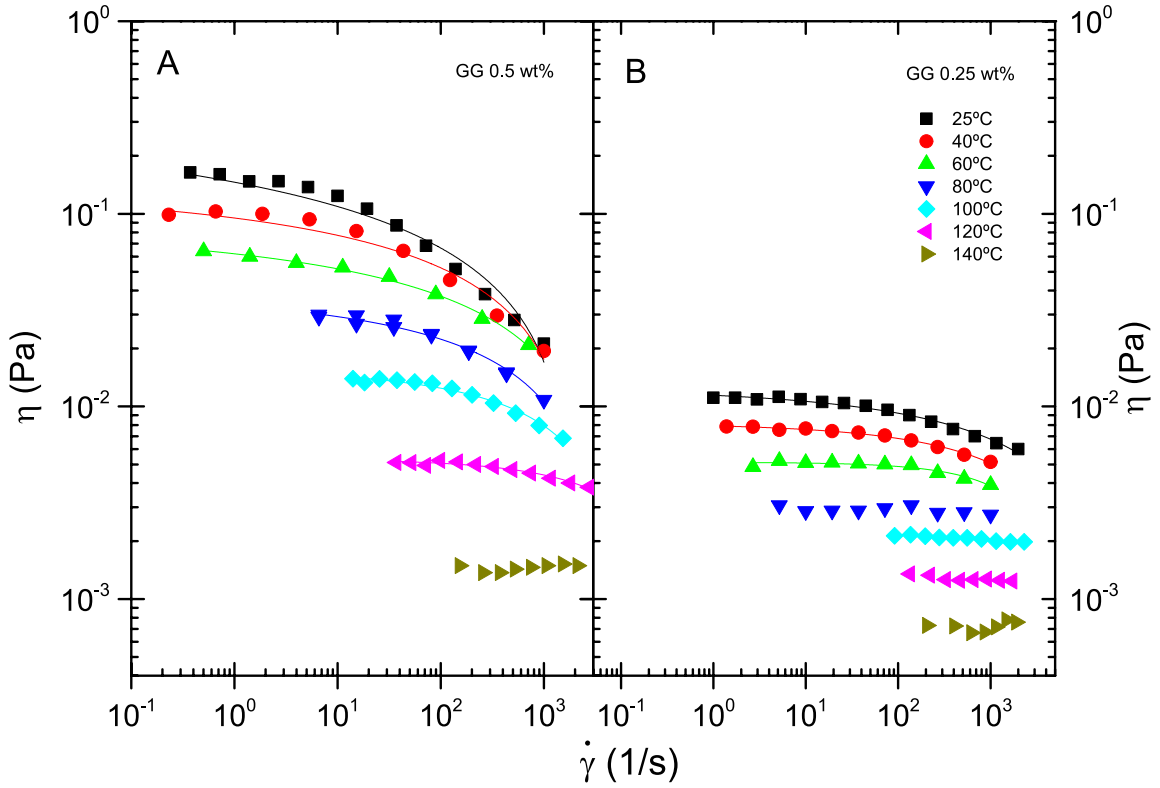

Fig. 7 Flow behaviour of GG solutions as a function of temperature. (A) 0.50 wt% GG. (B) 0.25 wt% GG

Table 2 Williamson's parameters of guar solutions as function of temperature

Temperature (ºC)	Guar content (wt%)					
	0.25			**0.50**		
	η_0(Pa·s)	K (Pa·s^n)	n	η_0 (Pa·s)	K (Pa·s^n)	n
25	0.0129	1.06E-3	1.24	0.378	0.232	1.06
40	0.082	2.6E-4	1.35	0.139	0.0445	1.14
60	0.0051	1.0E-5	1.69	0.086	0.024	1.15
80	0.0030	-	-	0.039	0.0057	1.23
100	0.0013			0.0157	5.8E-4	1.37
120	7.7E-4			0.0053	2.2E-5	1.53
140				0.0013		

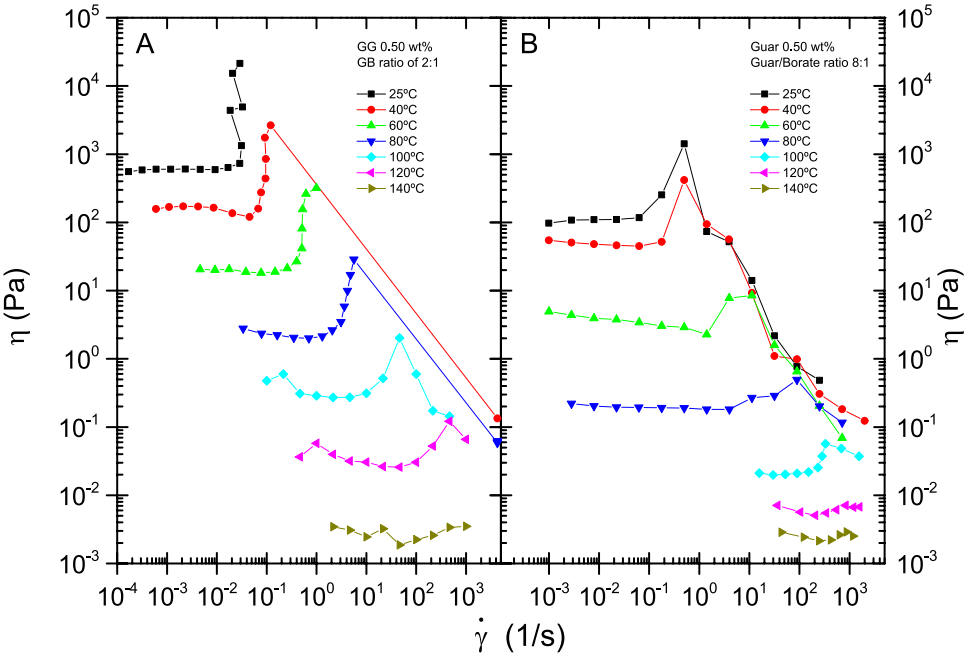

Fig. 8 Flow curves as a function of temperature for GB gels prepared with 0.50 wt% GG solution, at different GB ratios. (**A**) GB ratio of 2:1. (**B**) GB ratio of 8:1

$$\eta = \eta_0 - k \cdot \dot{\gamma}^{n-1} \quad (2)$$

where η is the apparent viscosity, η_0 is the zero-shear viscosity, k is the consistency index and n is the flow index. Table 2 lists the fitting parameters used for the Williamson model for GG solutions at different temperatures. As can be seen from this table, both the zero-shear viscosity and the consistency index decrease with increasing temperature for both GG concentrations. This trend in both parameters indicates that guar solutions progressively become more Newtonian-like at higher temperatures. In addition, it can be observed that n tends to increase with increasing temperature for the entire range studied.

Figure 8 depicts the viscosity flow curves of gels prepared with 0.50 wt% GG and GB ratios of 2:1 and 8:1 in the range of temperatures from 25 to 140ºC. In the case of gels formulated with 0.50 wt% GG, the addition of borate produced a significant increase in viscosity. Higher viscosities at low shear rates can be observed when the borate content increases (GB 2:1 ratio). It can be noticed that the viscosity values are significantly higher than those of the GG solution even at the highest shear rate for these fluids. The shear thinning observed at high shear rates is independent of borate concentration. Likewise, these gels exhibit a clear shear thickening at low shear rates with a maximum viscosity value. This complex flow behaviour of borate gels is essentially due to the microstructure developed from the borate ion complexation and crosslink bonds between monoborate ions and *cis*-hydroxyl groups (2:1 complexes) of the GG [24–26]. It is worth mentioning that this flow behaviour seems to be similar to that of associating polymers [50] and shear thickening polyacrylamide solutions [51].

The shear thickening effect observed in these gels could be attributed to the shear induced reorganization of the polymer associations during shearing. Thus, initially, increasing the shear rate elongates the polymer chains allowing an increased number of sites to be available for cross-link formation, which would form further junctions, and consequently raises the viscosity [52–54]. It has been stated that shear thickening in these gels depends on both shear rate

and shearing time [55]. For shear rates comparable to the relaxation time, shear thickening is caused by the increase in crosslink density. At higher shear rates and shorter times of shearing, the main mechanism is the increase in chain junction density with an important contribution of the non-Gaussian chain stretching at longer shearing times.

At higher shear rates, a significant decrease in viscosity is due to shear breaking of the bridges between the chains; consequently, this gives rise to a breakdown of the network structure. In addition, flow instabilities may also appear at high shear rates and longer times of shearing [56]. As temperature increases a significant drop in the viscosity is observed for both guar gels, more evident for high GB ratio. In addition, the critical shear rate at which shear thickening occurs is displaced toward higher shear rates with temperature. These notable changes in rheological response could be principally related to the decrease of borate ion conversion as the temperature is elevated [57]. This means that fewer borate ions are available to form 2:1 complexes with cis-hydroxyl pairs on the galactose side chains of GG, and therefore a weaker interconnected network is developed. Fewer borate ions in solution available to crosslink the guar chains, in addition to the reduction of the intensity of the intermolecular interactions due to the increase in temperature result in a viscosity decrease. Hence, when polymer chains are stretched and aligned during shear flow, a lower viscosity peak is observed. Furthermore, the gel with lower borate concentration shows a shift of the shear thickening effect to intermediate shear rates with a lower viscosity maximum value. This result reflects that the change in the temperature dependence of the borate ion conversion, alters the number of inter-chain junctions and, consequently, the maximum viscosity.

In the case of the gel with GB ratio of 8:1, at temperatures of 100ºC and above, a Newtonian plateau with similar zero-viscosity value to that of the guar solution is observed. This result suggests that the borate ion content is so low that the viscous behaviour is mainly controlled by the guar polymer interactions instead of by the borate associations. The reason for the shifting of the shear thickening phenomenon can probably be attributed to the higher shear rates required to fulfil the alignment and reorganization of polymer chains for intermolecular crosslinks at this higher temperature [58].

In Fig. 9, the steady-state flow curves for gels formulated with a guar solution at 0.25 wt% and different guar-borate ratios as a function of temperature are shown. Both gels show much lower viscosities than gels formulated using 0.50 wt% of guar, at a given temperature, over the whole range of shear rates studied. The rheological behaviour of both crosslinked gels containing 0.25 wt% guar is also very complex showing shear thickening and a maximum viscosity value with similar shear thinning behaviour at high shear rates. The main effect of decreasing the guar content is the decrease in viscosity.

As temperature increases, both GG 0.25 wt% gels behave as Newtonian fluids with either the same or slightly higher viscosity than the native GG solution. The range of temperatures where shear thickening appears is shortened. For 0.25 wt% GG gel at 8:1 GB ratio, the flow behaviour is almost the same as that of GG solution at temperatures above 80ºC. A lower concentration of biopolymer leads to a weaker network that shows mechanical properties very similar to the native solution.

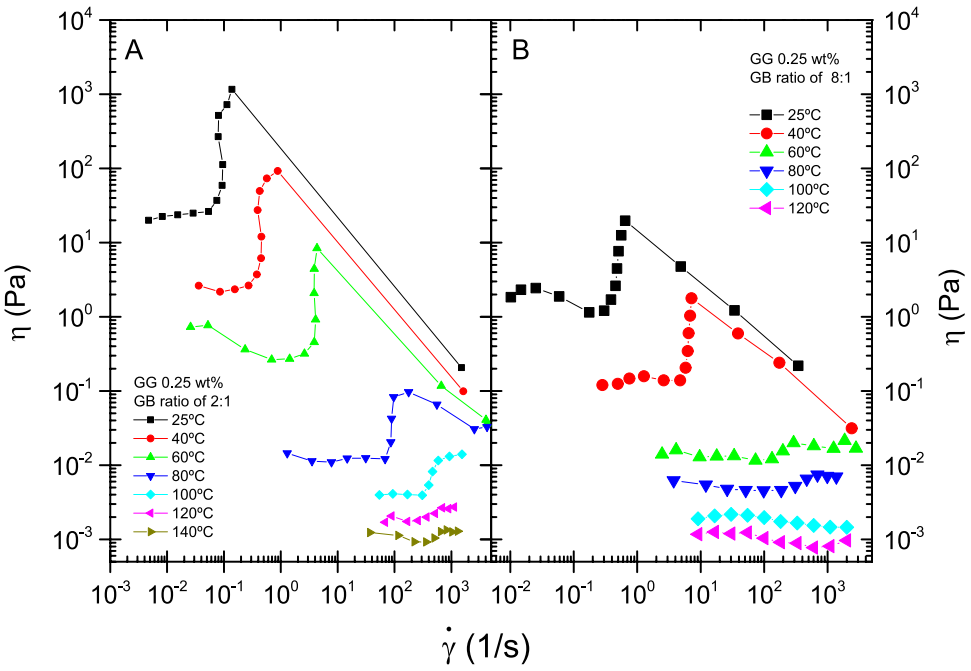

Fig. 9 Flow curves as a function of temperature for GB gels prepared with 0.25 wt% GG solution, at different GB ratios. (**A**) GB ratio of 2:1. (**B**) GB ratio of 8:1

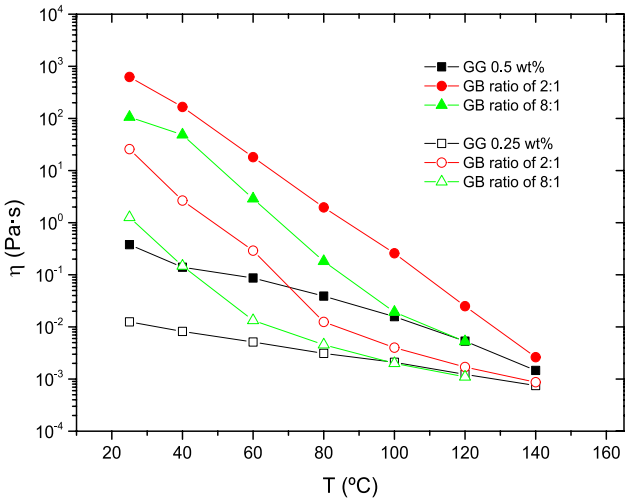

Fig. 10 Evolution of Newtonian viscosity for GG solutions and GB gels as a function of temperature and GB ratio. Solid symbols: 0.50 wt% GG and GB gels with ratios of 2:1 and 8:1. Open symbols: 0.25 wt% GG and GB gels with ratios of 2:1 and 8:1

The evolution of the viscosity of the GB gels as a function of temperature, measured at the Newtonian plateau, is shown in Fig. 10. Additionally, the zero-shear viscosity of the uncrosslinked guar solution is shown in this figure for comparison purposes. In the low temperature region, a significant increase in viscosity with respect to the guar solution is observed with both guar and borate concentrations. The gel formulated with GG 0.50 wt% and GB ratio of 2:1 shows higher viscosity values and greater thermal susceptibility. Nevertheless, the values of viscosity are higher than those of the GG solution up to 120ºC. At this GG concentration, a decrease in borate content leads to a decrease in viscosity at each temperature and a decrease in the resistance of the gel in the temperature range of 25-100ºC, but the slope of the thermal evolution of viscosity does not change significantly with borate concentration. The decrease in guar concentration leads to a significant decrease in the mechanical properties of the gel and its resistance to temperature. The gap between the viscosity of the gel and that of the guar solution is progressively reduced, achieving a residual value at higher temperatures, more evident for higher guar-borate ratios or lower borate content.

It is worth pointing out that the decrease in viscosity with temperature of these gels follows the same trend as that exhibited for the guar gel at high temperature. Consequently, there is a threshold temperature that depends on both borate and guar concentration where the number of borate bridging sites is so small that the rheological behaviour of these systems is governed by the polymer solution [29].

Conclusions

This study presents findings concerning the processing and rheological behaviour of guar-borate-crosslinked gels, with particular emphasis on their properties at temperatures exceeding the boiling point of the solvent. The processing of these gels was investigated using a rheo-reactor, characterising two stages: an initial mixing phase, followed by a reaction phase. The mixing time is primarily influenced by the viscosity of the GG solution (more concentrated solutions require longer mixing times). For a given GG solution viscosity, the mixing time varies only slightly with borate concentration. Following mixing, the complexation reaction is completed within 6 min, and even more rapidly at higher borate concentrations.

Both gel behaviour and gel strength decrease exponentially with temperature, with the effect being more pronounced at lower biopolymer and borate contents. However, the gel formulated with 0.5 wt% GG and a GB ratio of 2:1 retains its elastic properties up to 100°C. Above this temperature, thermal degradation leads to a discontinuous gel phase in which viscous properties dominate the rheological response.

The flow behaviour of GG solutions, as described by the Williamson model, is characterised by a Newtonian viscosity at low shear rates and pronounced shear thinning at high shear rates. Borate crosslinking increases the viscosity and introduces additional non-Newtonian features, which are governed by the concentration of available borate ions, polymer concentration and temperature. At low shear rates, a high-viscosity Newtonian region persists; at intermediate shear rates, a shear-thickening regime appears, followed by shear thinning at higher shear rates. Both the Newtonian viscosity and the shear-thickening behaviour are highly sensitive to temperature, guar concentration, and GB ratio, thereby limiting the effective upper temperature range for application in oil and gas recovery. This phenomenon is attributed to the combined effect of reduced crosslinking density and thermal degradation of the polysaccharide.

This study provides new insights into the influence of elevated temperatures on the rheological behaviour of guar-borate gels. However, it does not consider the combined effects of temperature and pressure, which are relevant in oil and gas recovery operations. Future research will focus on modelling to better understand how the interplay between composition, pressure, and temperature influences phase transitions and rheological performance in guar-borate systems.

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Author Contribution M.J.M.A. conceived the study, curated the data, conducted the formal analysis and investigation, developed the methodology, and wrote the original draft. F.M.B. contributed to the conceptualisation, formal analysis, and methodology; managed the project, supervised the work, and contributed to validation, visualisation, and manuscript review and editing. P.F.L. participated in the conceptualisation, formal analysis, and review and editing of the manuscript. All authors reviewed and approved the final version of the manuscript.

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Data Availability The datasets generated and analysed during the current study are available from the corresponding author upon reasonable request.

Declarations

Competing Interests The authors declare no competing interests.

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