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# A study of relation between suspension behavior and microstructure and viscoelastic property of guar gum fracturing fluid



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#### **ABSTRACT**

As a thickening agent, polysaccharide guar gum has been widely used in stimulation treatment. The viscosity is commonly used to evaluate the suspension properties. To fully characterize the proppant suspension behavior of guar gum solutions or gels, the microstructures and viscoelastic properties of hydroxypropyl guar gum (HPG) and carboxymethyl guar gum (CMG) were studied, respectively. Smallamplitude oscillatory shear (SAOS) measurements show that HPG gel has a higher elastic modulus than CMG gel with an identical apparent viscosity to the base fluid. According to the micrographs, the HPG fluid has a greater degree of association or overlap that composes a network structure than the CMG gel. Then, these samples were evaluated for their proppant suspension behavior using static-settlement tests. The results show that the proppant suspension properties of the crosslinked gels correlate with the microstructure and elasticity of the fluids. The HPG gel has a better suspension ability than the CMG gel.  $\odot$  2014 Elsevier B.V. All rights reserved.

## 1. Introduction

As a type of polysaccharide, guar gum is obtained from the seed endosperm of the guar plant called cyamopsis tetragonolobus ([Vijayendran and Bone, 1984\)](#page--1-0). Galactomannan is a highmolecular-weight water-soluble polysaccharide that consists of galactose and mannose. Because of its thickening ability, texture modifiability, good biodegradability and biocompatibility, polysaccharide has been widely used in industries such as polymer composites, foods, and cosmetics as well as in pharmacies. Furthermore, guar gum is the most popular polymer applied to prepare water-based fracturing fluids [\(Risica et al., 2010; Wang](#page--1-0) [and Zhang, 2007; Lei and Clark, 2004\)](#page--1-0).

The aqueous solution and gel of guar gum have been extensively used in hydraulic fracturing fluids. To transport the proppant along the length of the fracture and maintain it in suspension are the predominant functions of the fluids ([Goel et al., 2002](#page--1-0)). HPG and CMG derivatives have been used and investigated for decades (Castro [Castro Dantas et al. \(2003\)](#page--1-0)). Currently, guar fracturing fluid is usually characterized for its viscosity, which is used to determine its proppant suspension behavior ([Horri et al., 2011\)](#page--1-0). However, based on the static proppant test, HPG and CMG solutions with identical apparent viscosity were found to have different

<http://dx.doi.org/10.1016/j.petrol.2014.09.016> 0920-4105/© 2014 Elsevier B.V. All rights reserved. abilities to suspend proppant. Consequently, some additional factors, such as the crosslinked bond of the network structure, may also affect the suspension capabilities of the fluid. Thus, the viscosity could not accurately depict the network structure formed in the guar gel ([Harris and Heath, 1998; Kramer et al., 1987\)](#page--1-0). To accurately describe the proppant suspension capacity of a guarbased fracturing fluid, the microstructures and viscoelastic properties of these fluids were studied. Based on these studies, we can optimize the fracturing fluid selection and the performance and provide facts and figures to stimulate a formation. This study is notably significant for the improving stimulation effect.

#### 2. Experiments

### 2.1. Materials

The fluids, including HPG and CMG, were prepared as noncrosslinked linear solutions. A crosslinked gel was obtained by adding organic borate into the linear solution. All samples were prepared in Chengdu City tap water. HPG, CMG, borate crosslinker and  $NH_3\bullet H_2O$  were used as received from commercial sources without purification.  $NH_3\bullet H_2O$  was used to adjust the solution pH. This research focused on which fact contribution mainly to suspension the proppant in the fracturing process. Thus, the concentration of thickener was identical to that in the field.

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Although the fluid system included some other additive agents, they did not affect to the suspension ability and were missed in the study.

The used ceramic proppant is completely spherical and had a sieve size distribution of 20–40. The specific gravity was 2.41  $\rm g/cm^3$ .

#### 2.2. Sample preparations

First, 0.5 g of HPG was weighed and slowly incorporated into 100 ml of tap water, and the solution was stirred at 25  $\degree$ C for one hour for good hydration [\(Gao et al., 2008\)](#page--1-0), after which its pH was adjusted to 11 with  $NH_3\bullet H_2O$ . Then, 0.49 CMG was weighed, and the preparation process was repeated. These solutions have an equal apparent viscosity of 75 mPa s at 170 s $^{-1}$ . Through a micro syringe, the 0.5-g borate crosslinker was added, respectively into 100 ml of HPG and CMG solutions. These two solutions were thoroughly mixed by manually stirring with a muddler for approximately 10 s and subsequently set aside for several minutes to obtain an HPG and CMG gel. Then, these samples were characterized at ambient temperature for their rheology and microstructure.

A FEI Quanta 450 field emission environmental scanning electron microscope (ESEM) was used to observe the microstructures of the samples. A freeze drier was used to freeze the samples in liquid nitrogen in a vacuum environment. Then, they were glued on an aluminum stub, and the surfaces were coated with gold.

#### 2.3. Viscoelastic measurements

The viscoelastic properties of the fluids were obtained from SAOS measurements, which were performed on a controlled stress rheometer Thermo Scientific HAAKE MARS III equipped with a parallel-plate geometry. The measuring strain was 1%. The frequency was between  $10^{-2}$  and 100 rad/s, and all measurements were performed at  $25$  °C. The viscoelastic measurements were affected by the stress history imposed on the specimens before the measurements; thus, after loading in the rheometer, the samples were maintained at rest for a fixed time before starting these measurements. All the datas given were means of 3 tests.

### 2.4. Static-column test

The static-settling behaviors of the fluids were measured using a static-column test with a 11 glass cylinder. The testing fluids were measured using a 20/40 mesh proppant. The proppant was mixed into the beaker similarly to how such fluid would be mixed on location. Then, the fluid/proppant mixture was placed in a graduated cylinder, and the time for the proppant to settle was recorded. For this measurement, the total setting distance was 31 cm, and the total fluid volume was 1 L. The proppant level was monitored. A 100% suspension was defined when there was no decrease in proppant level; 0% suspension was defined when all of the proppant completely settled. This method provides insight into the settling behavior at low-shear conditions or after pumping terminated.

## 3. Results and discussion

#### 3.1. Viscoelastic measurements

Fig. 1 shows the behavior of the moduli  $G'$  and  $G''$  as a function of the angular frequency for HPG and CMG fluids. In Fig. 1A, the HPG solution has a identical to higher G' value than the CMG solution in the entire frequency range, which may result from the higher entanglement-structural strength of HPG. There is a crossover point between the two moduli, and the corresponding frequency is called the relaxation rate (denoted as  $\omega_{G' = G''}$ ) in the Maxwell model [\(Yount et al., 2005\)](#page--1-0). The inverse of the crossover frequency can be regarded as the relaxation time (denoted as  $\tau_{G' = G''}$ ) of the network. The relaxation time indicates the elasticity of these fluids. Thus, the relaxation time can be used as an index for the number and strength of the temporary crosslink junctions. When the network quickly relaxes, a proppant suspended in the fluid can more frequently settle based on the viscous region of the fluid ([Loveless et al., 2011](#page--1-0)). [Table 1](#page--1-0) shows that HPG has a longer relaxation time than CMG, which indicates that the strength of the



Fig. 1. Oscillatory measurements of the guar gum solutions (A) and gels (B). The HPG loading is 5 g/L, and the CMG loading is 4.9 g/L at 25 °C.

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