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Material Characterisation

Scanning electron microscopy analysis of guar gum in the dissolution, gelation and gel-breaking process



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ABSTRACT

To better understand the guar gum (GG) working process, we divided it into three steps: dissolution, gelation, and gel breaking. Freeze-drying and scanning electron microscopy were used to examine the microstructures of GG in different states. The dissolution step could be divided into two processes: swelling and dissolving. The microstructures during these two processes were lined structures and random ribbon-like structures, respectively. Further, during the gelation step, the random ribbon-like structures transformed to crosslinked ordered structures. Finally, the ribbon-like structures were broken in the gel breaking step, but the orderliness was not broken. These findings are beneficial to the potential modification or design of GG and the improvement of its hydrogel performance.

1. Introduction

Guar gum (GG) is a water-soluble polysaccharide that originates from the endosperm of *Cyamopsis tetragonolobus*. It is a galactomannan that consists of a (1 → 4)-linked β-D-mannopyranosyl backbone partially substituted at O-6 with α-D-galactopyranosyl side groups, with a mannose to galactose ratio of about 1.6–1.8 [1,2]. GG is the most popular polymer-based thickening agent used in water-based fracturing fluids in the field of hydraulic fracturing [3–5]. Hydraulic fracturing treatments require a high-viscosity hydrogel to transport solid particles into the fracture, such that the cracks remain unfilled so that the reservoir fluids can flow to the well through the highly conductive fracture [6]. A high-viscosity GG hydrogel can be obtained by crosslinking GG and polyvalent hydroxyl complexes; borate and zirconate crosslinking systems are the most widely used for this process [7]. In particular, aqueous-based borate cross-linkers have been widely used. It is generally believed that the increase in viscosity of GG is due to the formation of borate/guar complexes, whereby borate links two guar units at adjacent *cis*-hydroxy groups on the mannose backbone [8]. The crosslinking mechanism for borax-GG is shown in Scheme 1; this scheme shows that an increase in the molecular weight and concentration of the polymer can affect its crosslinking because of the enhanced molecular interactions between polymer chains [9]. Breakers are very important to complete the life cycle of fracturing fluids. After the pumping is complete, the fluid should break back to the viscosity of water. Oxidizing chemicals, such as persulfates, act as primary gel breakers to break down the filter cake and regain the formation permeability or fracture

conductivity. These breakers presumably oxidize the polymer backbone, causing chain breakdown and facilitating fluid flowback [10].

Herein, to better understand the working process of GG, we divided the process into three steps: dissolution, gelation, and gel breaking. Further, we used freeze-drying and scanning electron microscopy (SEM) to examine the microstructures of GG in different states. Also, rheological data was obtained to verify the explanation of the GG working process provided.

2. Methods

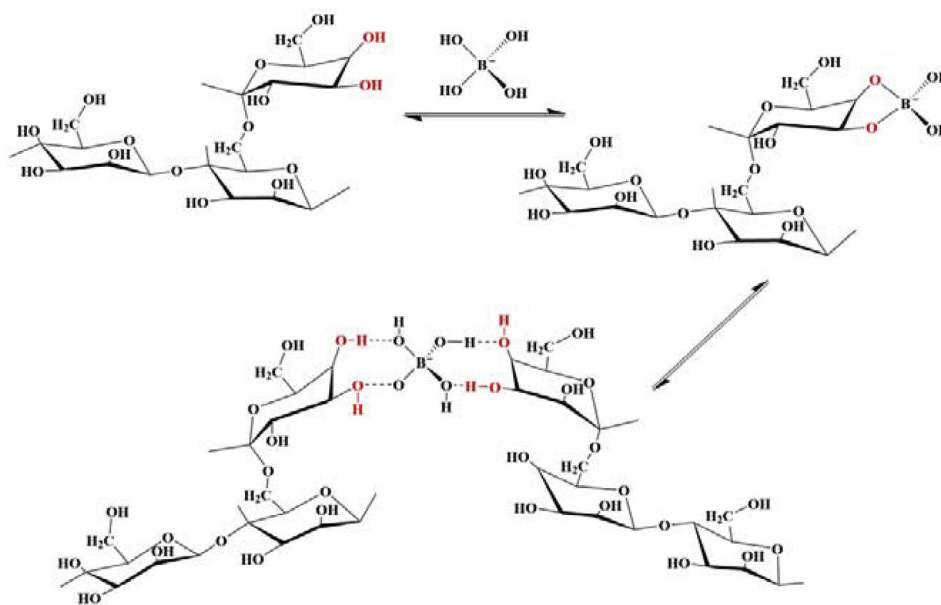
Solutions of 0.36 wt% GG (PetroChina Huabei Oilfield Company) and 4 wt% borax (Hongyan Chemical Reagent, Tianjin) were prepared. To obtain hydrogels, the above solutions were mixed in a ratio of 100:1 (v/v). In the gel-breaking step, ammonium persulphate (Hongyan Chemical Reagent, Tianjin), which accounts for 0.1% of the quality of the hydrogel, was added to the hydrogel. To observe the microstructures of GG during the dissolution, gelation, and gel-breaking steps, the GG solution, hydrogel, and gel-breaking liquid were treated in a freeze-drying machine (Songyuan Huaxing Technology, Beijing), and the obtained product was examined by a scanning electron microscope (TESCAN, Czech).

The rheological properties of the GG solution, hydrogel, and gel-breaking liquid were determined using a rheometer (Nirun Intelligent Technology, Shanghai) with a shear rate of 50 s⁻¹.

Particle size distribution was determined using a Malvern 2600 laser diffraction sizer (Malvern Instruments Ltd, Worcs, UK).

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Scheme 1. Equilibrium of borate ion complexation with *cis*-hydroxy pairs on GG molecule, leading to gel formation.

3. Results and discussion

3.1. Morphology of GG powder

Fig. 1a and Fig. 1b show the micro-morphology of GG particles at different magnifications (200 × and 10000 ×, respectively). Fig. 1a shows that the GG particles had no fixed shape. Further, the particle sizes were between 10 μm and 150 μm, which agreed with the results obtained using the laser diffraction size, as shown in Fig. 1c. The SEM image with a magnification of 10000 × showed the surface morphology of the GG particles (Fig. 1b). The GG particles were covered with a “lamellar texture,” which was responsible for the dispersion behavior of the GG powder in an aqueous medium [11].

3.2. Dissolution of GG powder

The microstructure during dissolution was studied intuitively using SEM. Fig. 2a and b shows the micrograph of the GG dispersion, which was different from that of the normal GG solution. The GG dispersion was sheared in water for 1.0 min and underwent swelling but not dissolving. It was clear from Fig. 2a that the GG powder swelled to form structures filled with lining threads. Moreover, the diameter of these lining threads was less than 1 μm and their length was more than 100 μm (Fig. 2b).

Fig. 2c displays the micrograph of the GG solution, which was sheared for 1.0 min and left to stand for 0.5 h. Some of the lining threads observed in Fig. 2a and b were transformed into ribbon-like structures. However, some lined structures remained at this stage. When the standing time was increased from 0.5 to 3.0 h, the number of ribbon-like structures increased and no lined structures were observed

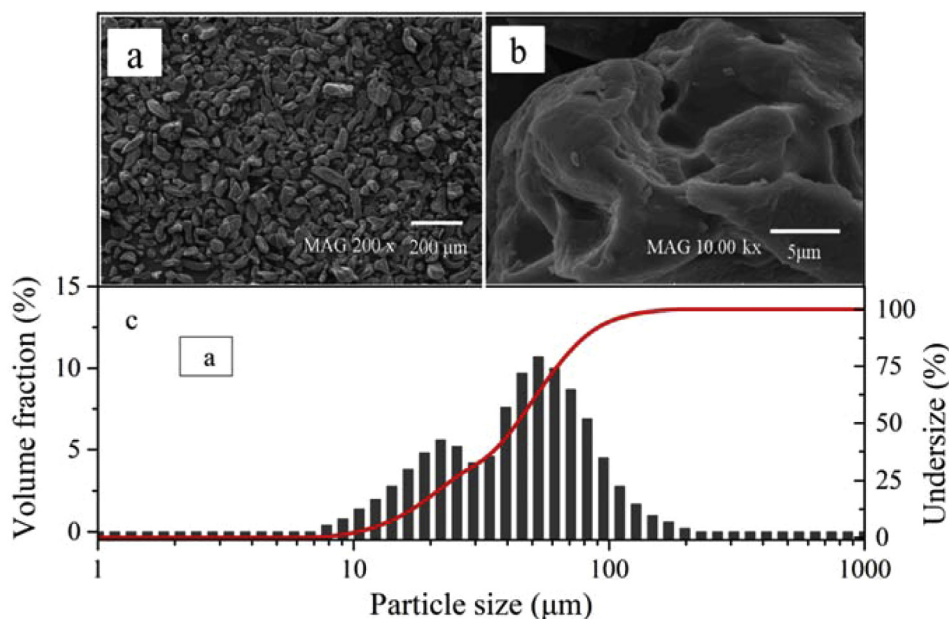


Fig. 1. Morphology and particle size distribution of GG.

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