

Synthesis of 2-alkenyl-3-butoxypropyl guar gum with enhanced rheological properties



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ABSTRACT

A new guar gum derivative was synthesized through the nucleophilic substitution of sodium hydroxide-activated guar gum with *n*-butyl glycidyl (BGE) ether. The physicochemical properties of 2-alkenyl-3-butoxypropyl guar gum (ABPG) were characterized by attenuated total reflection Fourier transform infrared spectrometry (ATR-FTIR), X-ray diffraction (XRD) and thermal gravimetric analyses (TGA). The results showed that sodium hydroxide can be effectively substituted with BGE to form the ABPG. The steady and dynamic rheological properties of the aqueous solution and ABPG gel were determined using an RS6000 rheometer. Compared with the guar gum, ABPG enhanced the thickening property and improved the solution stability. The ABPG gel exhibited good temperature resistance and shear stability properties.

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1. Introduction

Guar gum (GG) is a water-soluble polysaccharide composed of a linear chain of d-mannose residues connected by glycosidic linkages and can be obtained from the seeds of the legume *Cyamopsis tetragonolobus*. According to the structure of GG, (1 → 4)-linked β-D-mannopyranose units with α-D-galactopyranose units are connected to the mannose backbone through (1 → 6) glycosidic linkages. The ratio of mannose to galactose was approximately 2.6. Guar gum has been extensively used in various industrial applications [1,2]. It is a high molecular weight hydrocolloid hetero-polysaccharide composed of galactan and mannan units of approximately $2.8 \times 10^5 \text{ g mol}^{-1}$ molecular weight [3,4]. Due to its low cost, nontoxicity, and high water-solubility, guar gum possesses several attractive and industrially useful properties [5] and has been widely used in the pharmaceutical industry. The functional properties of GG are primarily of importance for controlling the release of drugs in the gastrointestinal tract. In the most common commercial use of GG additives, the additives act as carriers for colon-targeted drugs and anticancer drugs in the treatment of colorectal cancer [6–9]. GG has also been used in

preparing polyelectrolyte hydrogels with varying mechanical properties, swelling degree, and controlled drug release characteristics by chemically crosslinking GG with phosphatic agents [10,11] or polyacrylic acid [12]. As a fracturing fluid additive, GG has been used in mining and oilfield applications and plays important roles in enhancing oil recovery [13,14]. To improve the thickening and rheological abilities of guar gum, various modifications have been proposed [15]. These modifications introduce hydrophilic groups by etherification to increase water solubility and thickening ability. However, studies on hydrophobic modifications of guar gum have rarely been conducted. Modifications of guar gum with aryl groups may provide new functionalities and expand its applications. Guar gum derivatives modified with long chain hydrophilic groups have been synthesized and have many interesting properties, such as increased thickening ability and thermal stability and the ability to increase the shear resistance of fracturing fluids; however, these properties can be further improved with hydrophobic modifications [16,17]. The effects of hydrophobic modifications of guar gum on rheological properties were investigated to optimize hydrophobic-modified guar gum for use as a novel thickening agent in fracturing fluids.

In this work, for the first time, a new guar gum derivative containing alkenyl was synthesized through nucleophilic substitution. The modification reactions were systematically studied. The physicochemical properties of 2-alkenyl-3-butoxypropyl guar gum (ABPG) were characterized using ATR-FTIR, XRD and TGA. The

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steady and dynamic rheological properties of aqueous solutions and gels of ABPG were determined using a rheometer.

2. Material and methods

2.1. Materials

Powdered GG (2.61×10^5 g mol⁻¹ average molecular weight) was obtained from Renqiu ZhengHao Chemical Products Co., Ltd. Renqiu, Henan, China) and was used without further purification. The powdered carboxymethylated gum (CMG) was obtained from Renqiu ZhengHao Chemical Products Co., Ltd. (Renqiu, Henan, China). BGE was supplied by Sinopharm Group Co., Ltd. (Beijing, China). A crosslinking agent (JL-3) was purchased from Changqing Underground Additive Co., Ltd. (Xi'an, Shaanxi, China). Sodium hydroxide, ethanol, isopropanol and all other reagents used in this study were chemically pure and purchased from Beijing Chemical, Ltd. (Beijing, China).

2.2. Synthesis of ABPG

GG (20 g, 46 mmol) was completely dissolved in 40 ml isopropanol via stirring with a Teflon impeller at 300 rpm at 45 °C. At a constant temperature, sodium hydroxide (3.6 g, 92 mmol) was added under stirring at 300 rpm. The acid-base reaction was conducted under stirring at 45 °C for 2 h. After the pH was stabilized, BGE (12 g, 92 mmol) was gradually added to the solution. Etherification proceeded under stirring at 45 °C for 4 h. Then, the reaction mixture was cooled to room temperature. After centrifugation, washing, filtration and drying for 12 h at 60 °C (Fig. 1), the resultant light yellow powder of 2-alkenyl-3-butoxypropyl guar gum (ABPG) was obtained. ABPG were stored in a desiccator under vacuum until use.

2.3. Preparation of solution and gel

(1) Preparation of solutions:

Certain concentrations of GG and ABPG solutions were prepared. APBG (3 g) was gradually swollen in water (1,000 ml) with stirring at 300 rpm at 25 °C for 2 h to form a homogeneous mixture. The speed of the stirrer was carefully regulated. GG (5 g/L) was prepared under the same conditions. The solutions were stored in containers under room temperature until use.

(2) Preparation of gel:

Organic borate cross-linker JL-3 was added to 100 ml solutions of CMG and ABPG at a volume ratio of active solution to cross-linker of 100:0.3. The mixtures were stirred with a glass rod until homogeneous gels were formed.

2.4. Characterization of ABPG

The powdered GG and ABPG were pressed into KBr discs and characterized by FT-IR spectroscopy on a PerkinElmer Spectrum Two spectrometer (PerkinElmer Inc., USA) under dry air at room temperature. Powder X-ray diffraction (XRD) patterns of the synthesized products were recorded on a Rigaku model D/Max-B X-ray diffractometer (Brandt Instruments, Inc., Slidell, LA) at a scanning rate of 3°/min from 10° to 60°. Thermogravimetric experiments were performed on a Q500 TGA (TA Instruments, Inc., USA). Film samples approximately 5–10 mg were placed in a platinum sample pan and heated from 20 to 600 °C under an N₂ atmosphere at a heating rate of 10 °C/min.

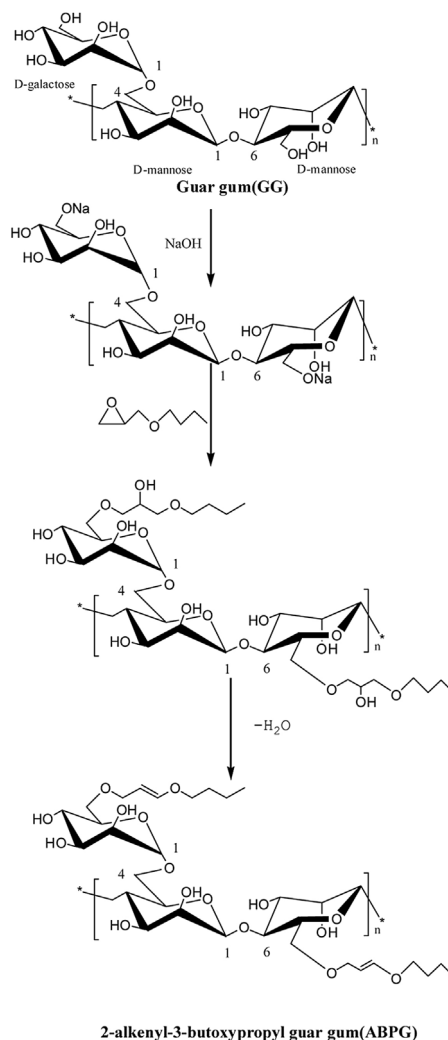


Fig. 1. The synthesis via of APBG.

2.5. Molecular weight estimation

The viscosity average molecular weights (M_v) of guar gum and ABPG were determined using intrinsic viscosity in the Mark-Houwink equation, $[\eta] = k M_v^\alpha$ with $\alpha = 0.732$ and $k = 3.8 \times 10^{-4}$. The molecular weight of a monomer unit of guar gum and ABPG was 270 as reported in the literature. Relative viscosity (η_r) was measured using an Ostwald capillary glass viscometer. The relative viscosity was used to determine the specific viscosity ($\eta_{sp} = \eta_r - 1$) from which the reduced viscosity $\eta_{red} = \eta_{sp}/C$ was determined. Intrinsic viscosity was generally determined by estimating the reduced viscosity at different concentrations in dilute solutions and extrapolating to zero concentration, i.e., $C = 0$.

2.6. Rheological property of solution and gel

The rheological properties of the GG and ABPG solutions and gels were determined using an American HAKER RS6000 rheometer (TA Instruments, Inc., USA) as follows:

- (1) The viscosity of ABPG solutions as a function of shear rate (0.1–200 1/s) was determined. The effects of ABPG concentration (3.0 g/L and 5.0 g/L), temperature (25 °C, 60 °C, and 80 °C), KCl concentration (10.0 g/L and 50.0 g/L) and pH (4.0 and 11.0) on viscosity were also examined.

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