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Preparation and Characterization of Hydroxypropyl Methacryloyl Guar Gum

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Abstract

Guar gum, derived from the Cyamopsis tetragonolobas plant, serves as an excellent and safe alternative to petroleum-based polymers. It is extensively used in various industries including the petroleum, textile, paper, food, and pharmaceutical industries. In this study, methacryloyl groups were introduced into hydroxypropyl guar gum to enhance its properties. This modification was analyzed using FTIR spectroscopy, which confirmed the structural changes that had occurred. Its thermal stability was assessed via TGA, while changes in its surface appearance and structure were analysed using SEM. The original fibrous particle structure of Guar gum undergoes a transition to a flake-like morphology due to the modification of Methacryloyl chains onto hydroxy Propyl guar gum. This indicates that the granular nature of guar gum is lost after derivatisation, resulting in the development of a flake-like structure.

Keywords: Guar gum, s, Derivatization, sustainable materials

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Introduction

Guar gum is a natural polymer obtained from the plant *Cyamopsis tetragonal bus*. It mainly contains 75–86% galactomannan, which gives it excellent thickening and stabilizing properties. Guar gum is of great importance in industries food, pharmaceuticals, textiles, and oil drilling, adsorbents, corrosion, because of its non-toxic, biodegradibity, low cost, and renewable origin. However, despite these advantages, its natural form has certain limitations including microbial degradation, susceptibility to enzyme degradation [1]. Guar gum can be modified by chemical Process including etherification [2], esterification [3] and cross linking [4] These modifications improve important properties such as solubility, clarity, thermal stability, and dissolution rate hydroxypropyl guar gum has already shown improved performance and is used in coal processing, detergents, and medical applications [5]. The active hydroxyl groups present in the structure of guar gum allows its modification through various chemical reactions. In this context, modification involving the methacryloyl group holds particular significance. Studies on the production of biodegradable polymeric films from methacryloyl-modified guar gum indicate that altering the chemical structure can lead to significant changes in the material's mechanical properties and surface morphology, thereby enhancing its performance and utility [6]. Chemically modified guar gum derivatives offer immense potential for application across various sectors due to their altered functional properties. They can be utilized in polymer composites [6-8], protective and functional coatings [9], textile processing [10], oil-field formulations [11], water treatment, and pharmaceutical systems [12]. Recent studies have expanded the utility of methacrylated guar gum beyond conventional applications to include the development of photo-crosslinkable hydrogels for 3D bioprinting, stem-cell delivery, and cartilage tissue engineering [13]. Thus, there is significant potential to develop methacrylated guar gum as a renewable, multifunctional, and chemically adaptable polymeric material. The Hydroxy propyl guar gum is modified to methacryloyl Hydroxypropyl guar gum for introducing double bold into the chain and enhance its performance for wider applications.

Materials and reagent

Hydroxypropyl guar gum with was obtained from M/s Lucid Colloids Pvt. Ltd., India, while methacryloyl chloride was purchased from Fisher Scientific and potassium hydroxide, Methyl ethyl ketone and Acetone were obtained from Qualigens Fine Chemicals. All chemicals used in the experiments were of analytical grade purity. The properties of hydroxypropyl guar gum with a DS of 0.4 (HPG4). It was found to contain 9–10% moisture and 4–6% ash. Its viscosity was measured to be 1400 cps at 30°C.

Degree of Substitution and Ester Value – The Ester value of the methacrloyl hydroxy propyl guar gum and Hydroxy propyl guar gum were evaluated as follows. A weighed samples of Hydroxy propyl guar gum and the derivatives were dissolved in 25 ml of 0.5 N NaOH and the ester allowed to hydrolyse completely over a Period of 15 hrs. Unreacted alkali was then back titrated against aqueous 0.2 N HCl from which the Degree of Substituition HPG and Methacryloyl Hydroxy Propyl Guar gum.

$$\text{Ester Value} = \frac{56100X\ Fuctionality}{Molecular\ Weight} \qquad (1)$$

Table 1. Variation of Reaction Time on Ester Value and Degree of Substitution of Modified Hydroxypropyl Guar Gum (M-HPG).

Sample name	Time of Reaction (Hrs)	Ester Value(mg KOH/gm)	Degree of Substituition
HPG	0	-	-
M-HPG 1	1	14.4	0.15
M-HPG3	3	22.2	0.22

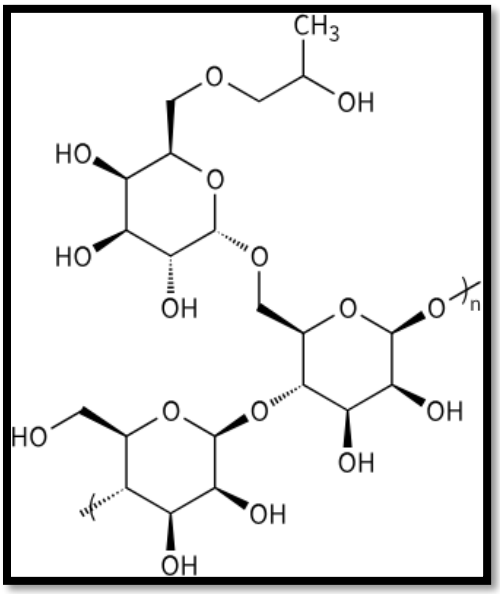


Figure 1 Chemical Structure of Hydroxy Propyl Guar Gum

Methacryloylation of Hrdroxy Propyl Guar gum - Prescribed quantity of Hydroxypropyl Guar Gum (HPG4 gm 80.9 mmoles) was thoroughly dissolved in a solvent mixture consisting of 50 mL distilled water and 50 mL Methyl Ethyl Ketone (MEK). To control the unwanted process of polymerization, 0.5 g of hydroquinone was added to the mixture. After this, triethyl amine was added as an acid absorber to neutralize the hydrochloric acid produced.

The prepared reaction mixture was transferred to a three-neck flask of 250 mL capacity. The flask was equipped with a mechanical stirrer, a thermometer to measure the temperature and a reflux condenser. The temperature of the reaction mixture was controlled between 0–5 °C with continuous mechanical stirring.

On the other hand, a separate solution was prepared by dissolving the required amount of methacryloyl chloride (8.46 gm 80.9 mmoles) in a mixture of 50 mL toluene and 10 mL MEK for the reaction. This solution was slowly flowed into the HPG reaction mixture drop by drop over a period of approximately 15 minutes while maintaining a temperature of 0–5 °C. The time when the methacryloyl chloride solution first entered the reaction mixture was determined as the initial time of the reaction.

The methacryloylation reaction was run continuously at a temperature of 5 °C for the prescribed period. After completion of the reaction period, the obtained methacryloyl hydroxypropyl guar gum (MA-HPG) was precipitated with the help of acetone. The precipitate obtained was separated by filtration and washed several times with acetone to remove residual reactants and soluble impurities.

Finally, the purified product was dried at a temperature of 50 °C and the drying process was continued until a constant weight was obtained. The completely dried methacryloylated HPG was stored in an airtight/sealed container for further physicochemical and structural analysis.

Characterization

FTIR : The structural analysis of Hydroxy Propyl Methacryloyl Guar Gum and Hydroxy Propyl Methacryloyl Guar Gum was conducted using a Nicolet 380 Attenuated Total Reflectance-Fourier Transform Infrared (ATR-FTIR) spectrometer. Measurements were performed in transmittance mode, with the samples prepared as KBr pellets and analyzed in absorbance mode.

TGA: Thermal stability and degradation behavior of the samples were evaluated using Thermogravimetric Analysis (TGA) under a nitrogen atmosphere. This test was conducted using a TA instrument, in which the sample was heated from room temperature to 600°C at a constant heating rate of 10°C per minute.

SEM: The surface morphology and micro-structural properties of hydroxypropyl methacryloyl guar gum were analyzed using Scanning Electron Microscopy (SEM). For this purpose, high-resolution micrographs were obtained using a Hitachi S-3700 microscope (Japan). Prior to analysis, the sample surfaces were coated with a thin layer of gold to enhance electrical conductivity and ensure the acquisition of clear, high-quality images. Subsequently, the samples were examined at high magnification to enable a detailed observation of surface micro-texture, structural characteristics, and morphological changes.

Results and Discussions

Fourier Transform Infrared Spectroscopy

Figure 2 presents a comparative analysis of the FTIR spectra of methacryloyl hydroxypropyl guar gum (MHPG 1&2). The broad and intense absorption band observed at 3420 cm^{-1} corresponds to the stretching vibration of hydroxyl (O–H) groups, while the prominent band at 2936 cm^{-1} is associated with the stretching vibration of C–H bonds. The absorption band appearing around 1640 cm^{-1} indicates the O–H bending vibration of bound water present in the sample, whereas the band at 1421 cm^{-1} corresponds to the bending vibration of the CH_2 group. Similarly, the band at 1025 cm^{-1} represents vibrations associated with the CH_2–O–CH_2 structural unit [13].

An additional absorption band appears at 1725 cm^{-1} in the MHPG spectrum, arising from the stretching vibration of the carbonyl (C=O) bond of the methacrylate group. The presence of this band provides significant evidence of the incorporation of methacryloyl groups into the guar gum structure. Furthermore, the band near 1635 cm^{-1} relates to the stretching vibration of the C=C bond, confirming the presence of the methacryloyl group [13]. With an increase in the amount of methacryloyl chloride, a decrease in the intensity of the O–H band and an increase in the distinctness of the C=O and C=C bands can be observed. These spectral changes indicate the chemical substitution of methacryloyl groups into the backbone structure of the guar gum.

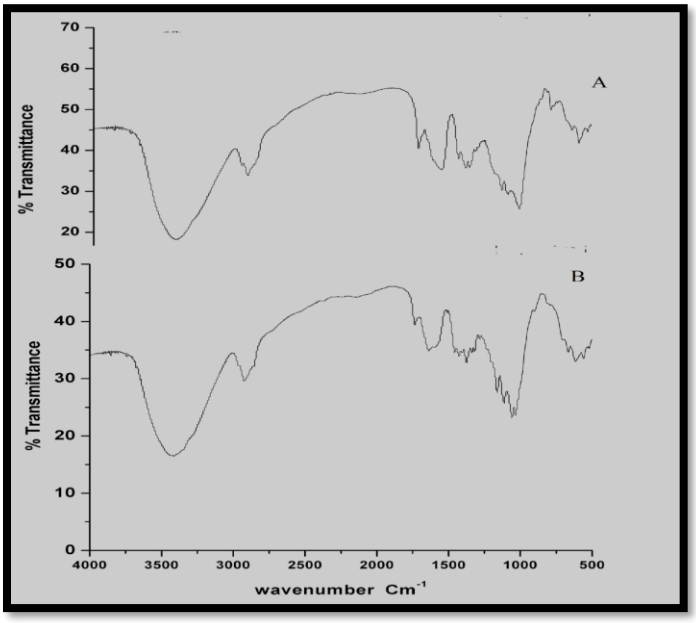


Fig 2: FTIR Spectrum of Methacryloyl Hydroxy propyl guar gum A (MHPG3) and B (MHPG1)

Thermogravimetric Analysis

TGA revealed that HPG showed two-step degradation with major weight loss between 240–340 °C, leaving 17.4% residue at 600 °C. MHPG derivatives degraded earlier, with single-step decomposition and higher residues (42.72–37.44%) at 600 °C. Increased substitution decreased thermal stability, as methacrylation reduced hydrogen bonding responsible for guar gum's partial stability.

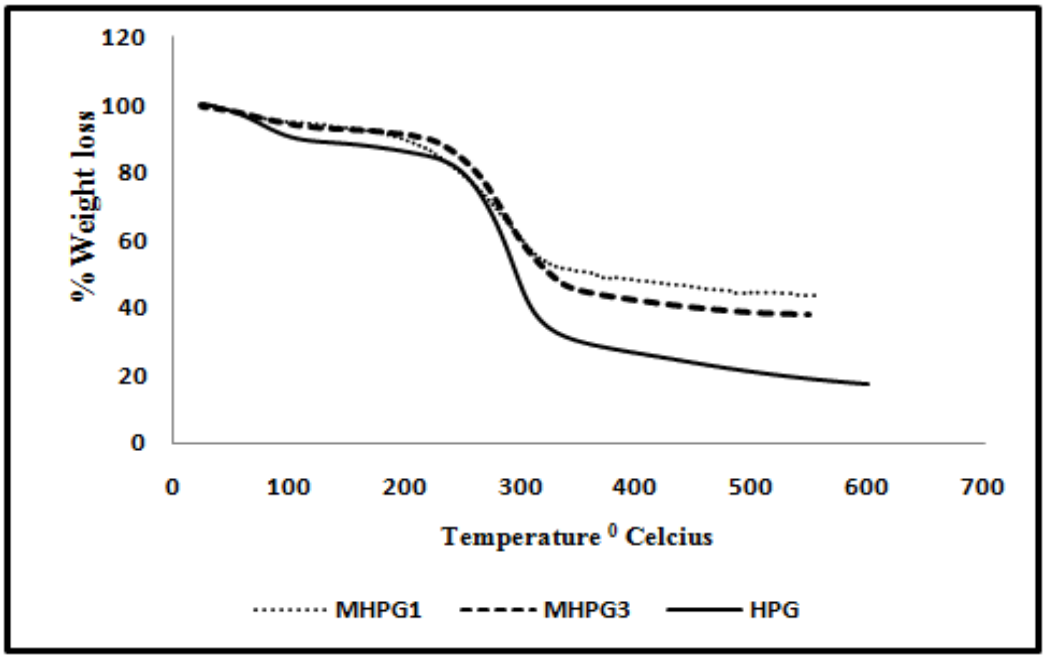


Fig 3: Thermogravimetric Analysis of Methacryloyl Hydroxy Propyl Guar gum and Hydroxy Propyl Guar gum

Morphological analysis

The surface morphology of hydroxypropyl methacryloyl guar gum was analyzed using a Scanning Electron Microscope (SEM), and the results are presented in Figure 4. Observations revealed that the surface of hydroxypropyl guar gum is fibrous in nature, whereas the surface of Methacryloyl Hydroxy Propyl Guargum (MHPG1) and (MHPG3) appears heterogeneous and irregular morphology [14]. This alteration indicates the successful derivatization of the guar gum.

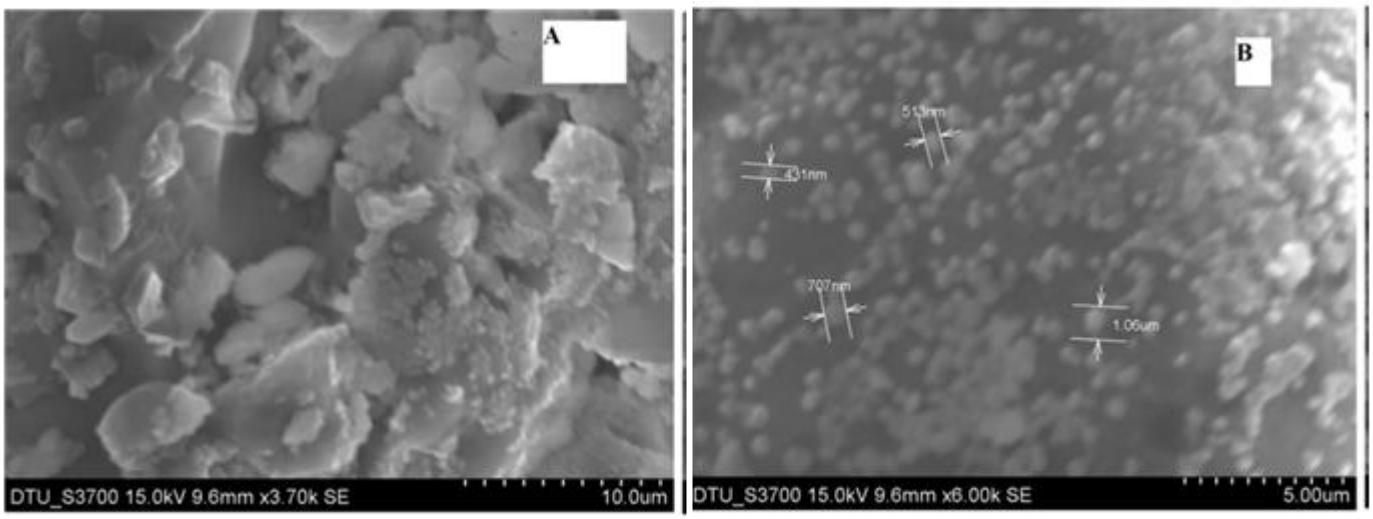


Figure 4 SEM micrographs showing the surface morphology and microstructure of methacryloyl hydroxypropyl guar gum (MA-HPG3): (A) image obtained at ×3.70k magnification with a 10 µm scale bar, and (B) Detailed surface texture at higher magnification, revealing nanoscale structural features ranging from approximately 431 to 707 nm in size.

Conclusions

In this study, methacryloyl-modified hydroxypropyl guar gum (MHPG) was synthesized by reacting hydroxypropyl guar gum (HPG) with methacryloyl chloride under controlled reaction conditions. The degree of substitution (DS) was investigated in relation to reaction duration to evaluate the key factors influencing the modification process. FTIR spectroscopy results revealed characteristic absorption bands associated with carbonyl (C=O) and carbon-carbon double bonds (C=C), confirming the incorporation of methacryloyl groups into the polymer structure. Additionally, scanning electron microscopy (SEM) images provided insights into changes in the surface morphology and microstructure of the modified sample. Variations observed in the degree of substitution and spectral characteristics indicated that the incorporation of methacryloyl groups could influence the chemical structure and surface properties of HPG. Future research involving detailed testing of its mechanical, thermal, biodegradable, and other functional properties could assess its potential utility in applications such as biodegradable films, protective coatings, hydrogels, and other advanced polymeric materials.

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