Preparation and Characterization of Hydroxy Propyl Allyl Guar Gum

Jay Prakash[1] ,Gopal Arora[1] and Swati Varshney[2]

[1]SBAS, Sanskriti University, Mathura, India
[2]Delhi Skill and Entrepreneurship University, Okhla-II Campus, Delhi

ABSTRACT

The primary objective of this work is to synthesize and characterize Hydroxy Propyl Allyl Guar Gum (HPAGG), a modified Guar gum derivative whose functional qualities are intended to be improved. To enhance its usefulness in a range of applications, hydroxypropyl and allyl groups were chemically added to guar gum during the preparation of HPAGG. Fourier Transform Infrared Spectroscopy (FTIR), which revealed important functional group transformations, was used to verify the structural integrity and successful modification. The derivative's thermal stability was assessed using thermogravimetric analysis (TGA), The process of modification resulted in morphological changes that were examined using Scanning Electron Microscopy (SEM). The thorough description highlights HPAGG's potential for cutting-edge applications in sectors like pharmaceuticals, agrochemicals and nanocomposites.

Key words:-Hydroxy propyl allyl guar gum, FTIR , SEM and TGA

1. Introduction: -

The seeds of the drought-tolerant Leguminosae plant Cyamopsis Tetragonalobus are utilized for the production of guar gum [1-2].75–86% water soluble galactomannan, 8–14% moisture, 5–6% protein, 2-3% fiber, and 0.5–1% ash is all present in powdered guar gum [3].Galactomannans show off a multitude of novel and practically useful properties. They make up the second largest group of polysaccharides and are present in the endosperm or cell walls of many plant seeds like Guar gum & cassia gum [4-7].
The fact that this guar gum is non-toxic and biodegradable contributes to its growing appeal [8]. Additionally, because it is affordable and derived from renewable natural resources, its supply can keep up with demand across a range of industries [9]. However, in more sophisticated technological applications, its inherent properties frequently restrict its functionality. When these raw gums are modified, hybrid derivatives are produced that retain the majority of the gums 'natural benefits while also adding new functional groups for desirable qualities [10]. Guar gum's hydroxyl groups make them excellent for changing the structure and functionalization of the material[11] so guar gum can be chemically modified to improve its physical and chemical properties and find a wider range of applications. For example, by forming their hydroxypropyl or carboxymethyl derivatives, guar gum solution's solubility, solubilization time, and clarity can all be significantly increased [12,13].Higher heat resistance and a quicker rate of dissolution are anticipated in hydroxypropyl guar gum when compared to pure guar gum, allowing

for uses as natural additives in coal, detergent, and the medical sector [14-17].Recently, HPG and related polysaccharides have been combined to create a range of very effective and adaptable materials with a variety of applications[18-19]. Here, hydroxypropyl and allyl groups are added to guar gum to produce a novel derivative called hydroxypropyl allyl guar gum (HPAGG). By enhancing its solubility, mechanical qualities, and thermal stability, this modification hopes to increase its potential in a variety of industries, including biopolymers, textiles, and pharmaceuticals. Using Fourier Transform Infrared Spectroscopy (FTIR), Thermogravimetric Analysis (TGA) and Scanning Electron Microscopy (SEM), this paper addresses the preparation of HPAGG and its comprehensive characterization

2. Materials and reagent

The hydroxypropyl guar gum were obtained from M/S Lucid Colloids Pvt. Ltd. India. and allyl Chloride was purchase from Fisher Scientific, isopropyl alcohol was purchase from Qualigens Fine Chemicals. all reagents in the experiments were of pure grade. Hydroxypropyl guar gum (DS-0.4) (HPG4)

3. Synthesis of Hydroxy propyl AllylGuar Gum (HPAGG): -

In order to achieve etherification, hydroxy propyl guar gum reacts with an allyl chloride in the presence of a water-isopropanol solvent mixture. This was accomplished by dispersing 10 grams of Hydroxy Propyl Guar Gum (HPG) flour solution to form an isopropyl solution in a clean, three-necked, one-litre round button flask that was kept at 40°C and purged with nitrogen after an hour. Next, a predetermined quantity of 50% (w/v) sodium hydroxide aq. solution and a quaternary ammonium salt (phase transfer agent) were added to the slurry, stirred, and heated to 50°C. The reaction solution was heated to 4 hrs (Table 2). The reaction mixture was cooled gradually dispersed in acetone and the excess of alkali is neutralized with glacial acetic acid bringing the ph to 7. The product than finally washed with acetone and then dried under vacuum.

Table-1 Physical Properties of Hydroxy Propyl Guar Gum

Sample	Hydroxypropyl Guar Gum (HPG4)
Moisture Content	9-10%
Ash Content	4-6%
Viscosity at $30^{O}C$	1400 Cps

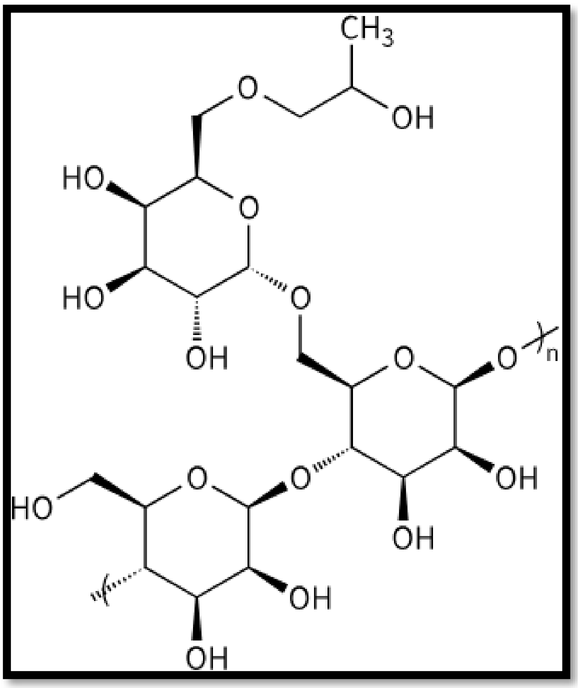

Fig-1 Chemical Structure of Hydroxy Propyl Guar Gum

Table-2 Reaction condition for the formation of Hydroxy Propyl allylGuar-gum

Sample Name	Volume (NaOH)	Weight (Allyl Chloride)	Time (Hrs)	Temperature °C
HPAG1	3	2.78	2	50
HPAG2	3	2.78	4	50

4. Characterization

FTIR : The Hydroxy propyl guar gum and Allyl Hydroxy Propyl Guar-gum was analysed by a Nicolet 380 Attenuated total reflectance-Fourier transform infrared (ATR-FTIR) Spectrometry in transmittance mode and the samples were analysed in absorbance mode using KBr pellets.

TGA: Thermal degradation was performed under a nitrogen atmosphere using TA instruments, with a heating rate of 10°C per minute, starting from room temperature up to 600°C.

SEM : Scanning Electron Microscopy (SEM) is a powerful technique used to examine the surface morphology and microstructure of materials at high magnifications. SEM images were captured using a Hitachi S-3700 microscope (Japan). The Hydroxy Propyl guar gum and Allyl Hydroxy Propyl guar gum samples were coated with a thin layer of gold, then observed and photographed.

5. Results and Discussions:

5.1. Fourier Transform Infrared Spectroscopy: -

Fig. 2 shows the comparison of spectra for Hydroxypropyl guar gum (HPG) and Hydroxy propyl allyl guar gum (HPAGG). The presence of a very strong and broad absorption band at 3420 cm^{-1} is assigned to OH bond stretching, while the sharp absorption band located at 2936 cm^{-1} may be attributed to CH group stretching. The absorption band appearing at 1640 cm^{-1} is due the OH bond belonging to water molecules. CH_2 group bending is assigned to an absorption band located at 1421 cm^{-1}, and the bending of CH_2- O-CH_2, appears in the 1025 cm^{-1} frequency region[20]. It was observed that on allylation a peak at approximately 1568 cm^{-1} appeared in the infrared spectrum of a Hydroxy propyl allyl guar gum corresponding to the stretching vibration of C=C it demonstrate the existence of allyl groups, respectively which may be attributed to the incorporation of allyl groups into the hydroxy propyl guar gum molecule from the FTIR results, it may observed that as the concentration of allyl chloride increases the peak intensity at 1650 cm^{-1}gets decreases and at 1568cm^{-1}increases.

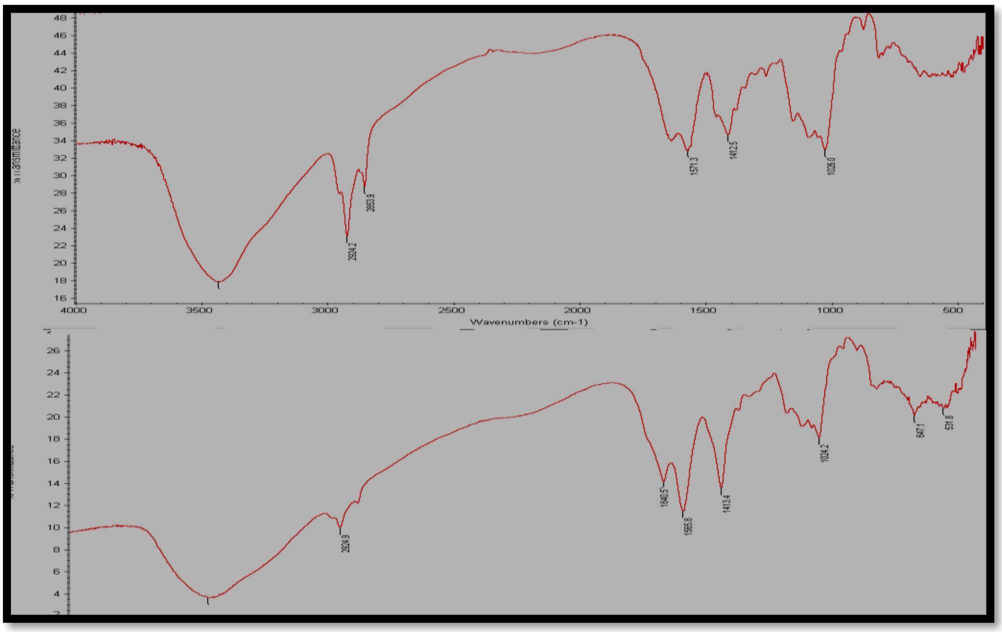

Fig-2 FTIR spectrum of (A) Hydroxypropyl guar gum (HPG) (B) Hydroxypropyl allyl guar gum (HPAG1)

5.3. Thermal analysis

The TGA curve of HPAGG are shown in Fig 3.It shows two distinct zone are observed where the weight is being lost. The initial weight loss is due to the presence of small amount of moisture in the sample. The second zone of weight loss is observed when the polymer decomposition takes place. The rate of weight loss is increased on increasing the temperature upto 200℃ but thereafter the rate of weight loss was found to decrease with temperature About 37% weight loss occurred between 200-300℃ and at 600℃ only 18.64%residue was obtained. In case of Hydroxy propyl allyl guar gum (HPAG1) 26.6 % residue was obtained while incase of Hydroxy propyl allyl guar gum (HPAG2) 30.03% residue was obtained So from the TGA results, we conclude that the incorporation of allyl group onto the Hydroxy propyl guar gum enhances the thermal stability of Hydroxy Propyl Guar Gum

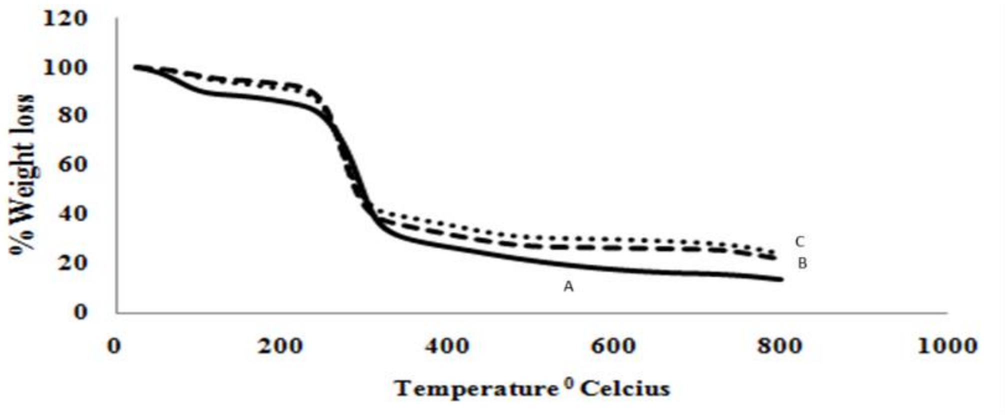

Fig 3: - TGA of (A) Hydoxy Propyl Gaur Gum (HPGG), (B) Hydroxypropy Allyl guar gum1 (HPAG1)(C) Hydroxy propyl allyl guar gum (HPAG2)

5.4. Morphological Analysis: -

Using a scanning electron microscope (SEM), the surface morphological study of hydroxy propyl guar gum and hydroxy propyl allyl guar gum was evaluated. The results are shown in Fig 4. According to the scanning result, Hydroxypropyl guar gum has a surface with fibrous structures, but Hydroxypropyl allyl guar gum is rougher and has an uneven form[21]. This could support the hydroxyl propyl guar gum derivatization.

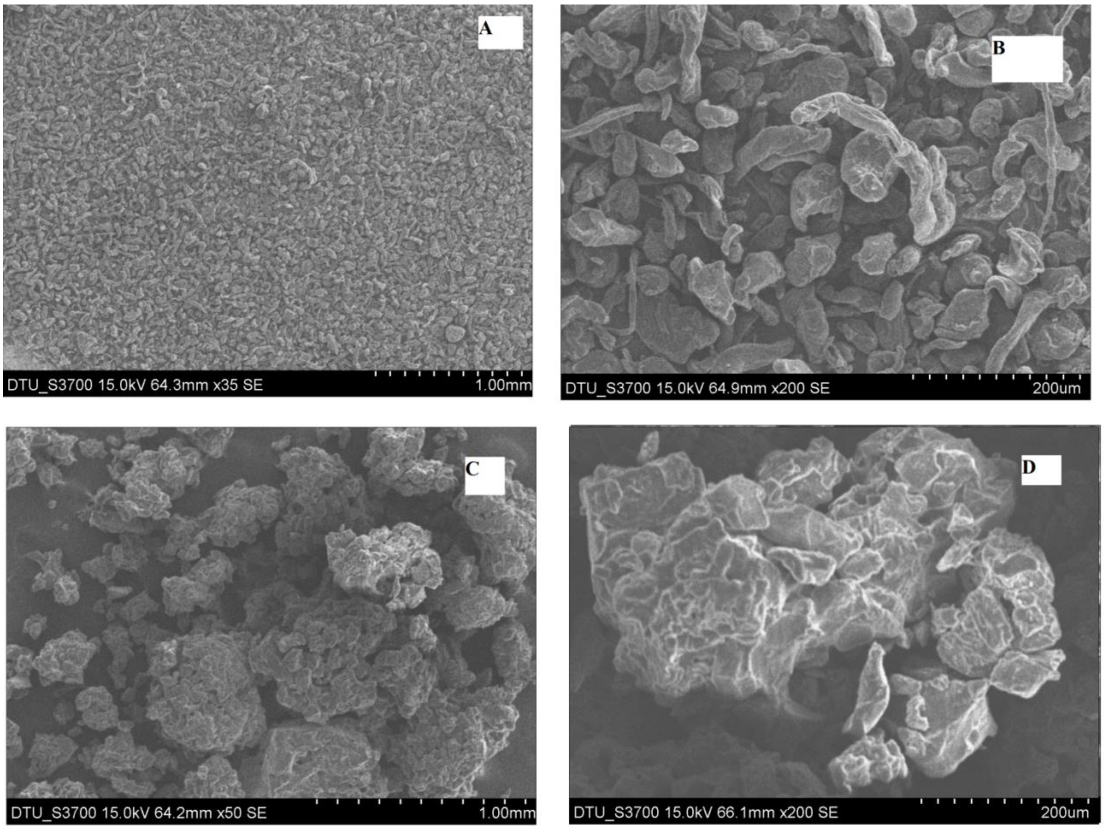

Fig 4:- SEM images of (A, B) Hydroxypropyl guar gum (HPG) (C, D) Hydroxy propyl Allyl guar gum (HPAG1)

5. Conclusions

Allyl modified Hydroxy propyl guar gum was efficiently prepared under moderate reaction conditions. From the characterization studies, it is being confirmed that the hydroxyl propyl guar gum has been derivatised with allyl chloride resulting in to a formation of a modified naturally occurring polysaccharide Hydroxy propyl allyl guargum.The continuation of this long-term study on gaur gum and the possibility of the emergence of numerous such novel derivatives in the future are both forward-thinking ideas.

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