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Iodine catalyzed acetylation of guar gum

Jay J.Patel*, Mandar Karve, Nirmal K.Patel

Department of Industrial ChemistryInstitute of Science & Technology for advanced studies & Research, V.V.Nagar 388120; Gujarat, (INDIA)

E-mail: drnirmalpatel@yahoo.com

ABSTRACT

Eaters of guar gum (GG) were synthesized by the solvent free method. Guar gum was acetylated with acetic anhydride and iodine as catalyst without any use of solvent. The reaction was carried out at 50^0C for 4 hrs. with stirring. The product is purified by acetone and the D.S. is measured. The D.S. of the product obtained was 0.6. The synthesized product was characterized by Fourier Transform Infrared Spectroscopy (FTIR).

KEYWORDS

Guar gum (GG);
Iodine;
Acetic anhydride;
Acetylation.

INTRODUCTION

Guar gum is galactomannan, derived from guar seeds (cyamopsis tetragonolobus) which belong to family *Leguminose*[1]. Its solution in water possesses high viscosity[2]. Due to these properties it finds its application in various industries viz. mining, textile, explosives, paper, petroleum, cosmetic and food industry[3]. Guar gum has better bioavailability and biocompatibility. Today, guar gum is also used as a controlled-release agent for the drug due to high hydration rate (Swelling in aqueous media). It also used as thickener and stabilizer in pharmaceutical formulation. When mixed with different ingredients in the formulation of tablets it form protective layer and consequently, drug releases out from the guar gum tablet in a sustained manner, achieving the desired kinetics effect, and masked unpleasant taste and odor of drugs and improve its stability and drug release properties[4-9]. Guar gum is also used as release modifier in sustain release tablet formulation[10]. Modified Guar gum is widely used in pharmaceutical application due to its viscosity increasing properties. The therapeutic effect of Guar gum is due to its ability to swell rapidly in aqueous media to form viscous dispersions or gels. When inhaled guar gum absorbed in the stomach and halted or alters absorption of glucose, cholesterol and possibly drugs[11-13]. Modified guar gum has a low swelling index and viscosity than guar gum. Due to these researchers nowadays focus on various derivatives of guar gum viz. O-(2-Hydroxyethyl), O-(2-hydroxypropyl) and O-carboxymethyl[14]. There are no. of methods are reported to modified natural polysaccharides, such as using acetyl chloride with pyridine,[15] carboxylic acids in situ activated with tosyl choride,[16] iminium chloride,[17] Na-salt of chloro acetic acid with NaOH[18] and acetyl chloride with $FeCl_3$as catalyst[19].

In the present study, we focused on esterification of guar gum, using iodine as catalyst. Hence the conventional reagent acetic anhydride was explored using iodine as catalyst. This esterification method has two advantages over other methods. First is the acylating reagent is used is not expensive than p-toluene sulfonyl

chloride, 1,1'-carbonyldiimidazole, etc. Second is the reaction carried out in homogeneous phase and no expensive solvent is used for the reaction.

EXPERIMENTAL

Materials and methods

Guar gum, acetic anhydride, sodium thiosulfate and iodine were purchased from Sigma- Aldrich. Solvent like acetone was of A.R. grade and use without any purification. All other reagents were of LR grade.

Acetylation of guar gum

Iodine (0.50 gm.) was added into pre-dried round bottom flask. Acetic anhydride (5 ml.) was added and the reaction mixture was stirred for 15 min. After that guar gum (1.0 gm.) was added to the mixture and the mixture was refluxed for 4 hrs. After the reaction was completed, excess iodine was removed by adding a saturated solution of sodium thiosulfate. Thus formed white precipitates of guar gum acetate was filtered off and washed thrice with cold water and then reprecipitated from acetone. The precipitates of guar gum acetate were dried under vacuum at 50^0C for 12 hrs.

Degree of substitution in modified guar gum

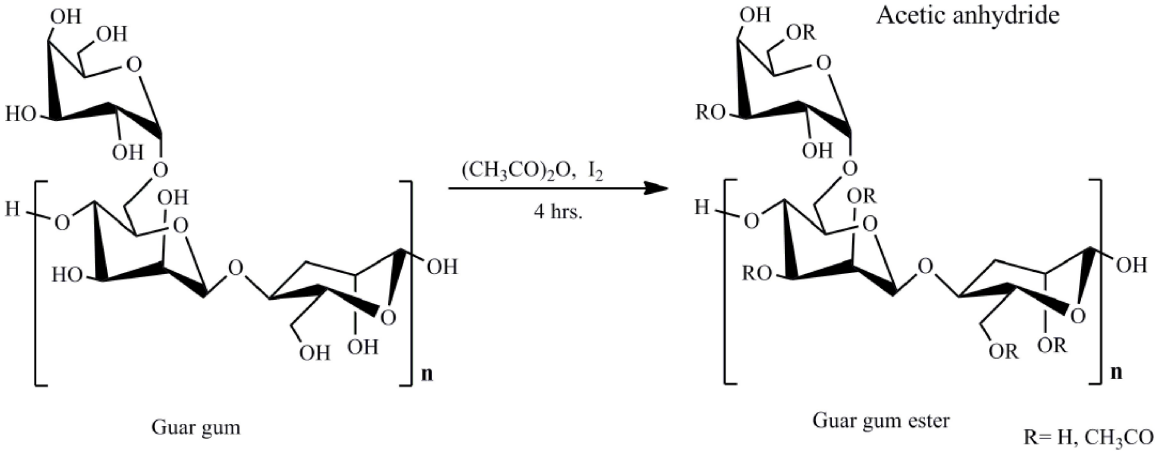
The D.S. of the product find out by method reported by N.K. Patel, et. al.[19]

1 gm. of CMGG was dissolved in known amount of water. Then this solution was passed through regenerated Amberlite IRA 96 anion exchange resin no. of times till it become acidic. Then the solution was divided into two equal parts labeled as solution 1 and solution 2. The exhausted resin was regenerated by passing 1 N HCl solution (3-4 times) followed by washing with distilled water to remove any excess acid.

Solution 1 was taken into previously weighed beaker. Evaporate water by heating on a hotplate and cool it into desiccator and weigh it again. Find the weight of residue left in the beaker. Find out concentration by evaporation.

Solution 2 was titrated against a standard solution of NaOH. Note down the burette reading and find out the degree of substitution by following equation.

$$DS = \frac{0.162\ B}{1 - 0.58\ B}$$



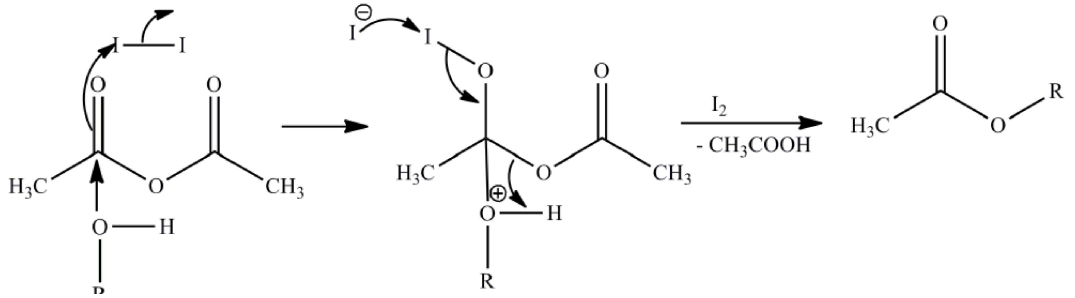


Figure 1 : Reaction mechanism

Where, $B = \frac{\text{Volumeof 1NNaOHused}}{\text{Weightofsample}}$

Characterization of modified guar gum

The resulting products were characterized by FTIR spectroscopy using Perkin Elmer spectrum GX instrument, by the KBr pallet method.

RESULT AND DISCUSSION

Acetylation of natural polysaccharides and its derivatives can be carried out using different acetylating agents as well as catalyst. Traditionally used catalysts such as aluminium chloride, sodium hydroxide required the use of costly solvent to carry out the reaction in homogeneous phase. But the use of iodine as catalyst can overcome the use of costly solvent also benefited by prevention of glycosidic linkage of polysaccharides ring which can be broken by traditional catalyst[20,21].

Reaction mechanism

An acid anhydride and an alcohol react together in

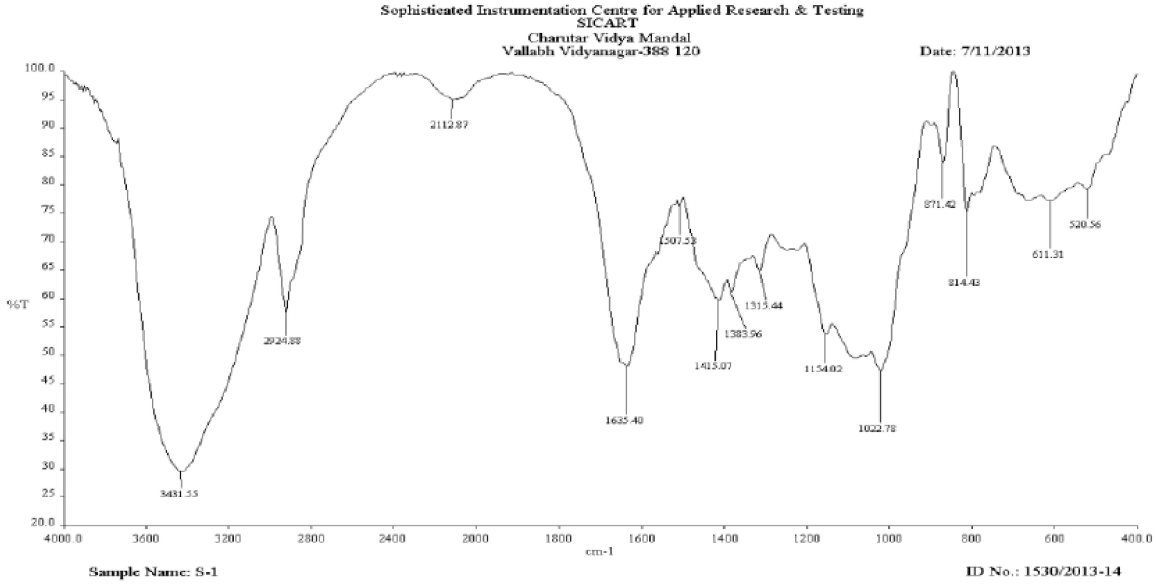


Figure 2 : IR spectra of GG

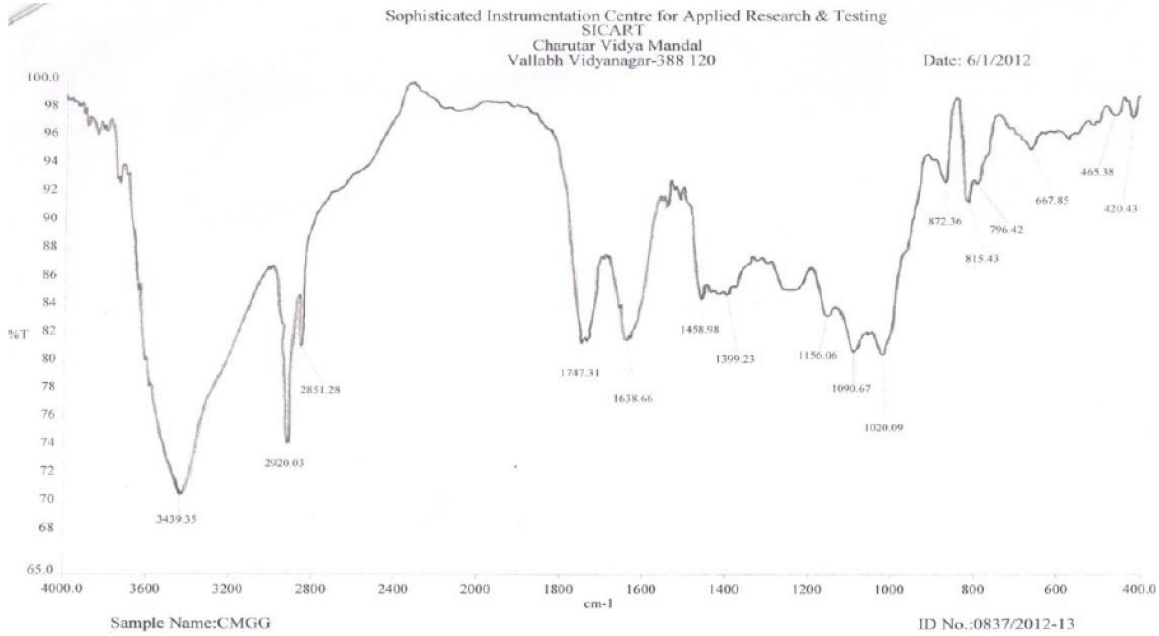


Figure 3 : IR spectra of CMGG

the presence of iodine as catalyst. The general mechanism is shown in Figure 1. In the first step acetic anhydride activated by iodine in the presence of –OH groups. The oxygen of R-OH attacks the carbonyl carbon resulting in sp^3 hybridization. The acetic ester is formed by the reaction of the iodine ion with the iodine atom that was attached to the oxygen of the carbonyl group, resulting in free iodine. Acetic acid is formed as a by-product. At the end of the reaction, addition of a saturated solution of sodium thiosulfate removes all the free iodine.

FTIR ANALYSIS

The IR spectrum of guar gum and acetylated guar gum was shown in Figure 2 and figure 3 respectively. The IR spectrum of acetylated guar gum shown a reduced intensity of the absorption band located at 3439 cm^{-1}, as compared to guar gum IR spectrum due to -OH is stretching, indicating that some -OH group were acetylated. The C-O symmetrical and asymmetrical and vibrations at a frequency of 1090.67 cm^{-1} and 1156.06 cm^{-1} confirms the incorporation of the acetyl group on to the guar gum molecule, which is absent in the guar gum spectra.

CONCLUSION

Acetylation of guar gum under solvent-free conditions using iodine as catalyst was carried out successfully. The D.S. of the product obtained was 0.6. The ester of guar gum is soluble in water.

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