Research Paper

Volume : 3 | Issue : 3 | March 2014 • ISSN No 2277 - 8179

Preperation and Characterization of Allyl Modified Guar Gum

Chemistry

KEYWORDS : Chemical Modification , Guar gum , Derivatisation

A.P Gupta
Professor, Department of Applied Chemistry & Polymer Technology, Delhi Technological, University, (Formerly Delhi College of Engineering), Bawana Road Shahbad Dairy, Delhi -110042

Gopal Arora
Research Scholar, Department of Applied Chemistry & Polymer Technology, Delhi Technological, University, (Formerly Delhi College of Engineering), Bawana Road Shahbad Dairy, Delhi -110042

ABSTRACT

The modification here discussed includes the introduction of allyl groups into the molecule for extending the application of guar-gum . The resulting product Allyl Guar –gum have been characterized by means of FTIR spectroscopy , Thermogravimetric analysis(TGA) and Scanning electron microscopy (SEM) . The invented product are superior to conventional gums , for application in petroleum , textile printing , paper , food and pharmaceutical Industries

Introduction

Rapidly growing demand for materials and products from renewable resources made it worthwhile to devlop new functional properties of guar –gum through physical or chemical modification , due to their suistainibility and biodegradability Guar gum is a non-ionic naturally occurring polysaccharide extracted from the endosperm of cyanopsis tetragonolobus and structurally is a galactomannan with a galactose to mannose ratio of approximately 1:2 .There has been some discussion regarding the distribution of the galactose on the mannose backbone , investigations have indicated that the distribution is not uniform but occurs in blocks with galactose rich and deficient regions [1] . The solution of which are highly viscous in nature. This property has allowed these polymers and their derivatives to be commercialized in field such as textiles [2,] ,Foods [3], Cosmetics[4] , Pharmaceuticals [5] and Oil recovery and drilling[6] . The solution blend of guar gum and polyvinyl alcohol were prepared and casting onto a teflon mold and evaporating the water at 40 0C in an air circulating oven [7,8]. Even if Guar gum and its derivatives are well known for wider application like other polysaccharides they evidence some drawbacks , such as uncontrolled release of hydration , pH dependent solubility , high susceptibility to microbial attack , chemical modification provides an efficient route for removing such drawbacks . Native guar gum can be modified into various water-soluble derivates by using reactive groups, to substitute the free hydroxyl groups along the macromolecule backbone upon dissolution in water, guar gum may give rise to as much as 10-14% insoluble residue, as depending on gum purity. The formation of such residues, mainly composed of heavily intertwined polysaccharide chains, proteins and ashes, together with the tendency of guargum to form aggregates in solution, are indesirable characteristics in some applications. Thus the gum is modified by One or more reactions such a depolymerisations [9] cyanoethylation ,quaternisation and sulphation[10] ,carboxymethylation[11] .Guar gum derivatives including acryloyl guar gum [12] , acetylated guar gum , [13] methacryloyl guar Gum [14], Guar gum methyl ether [15] are studied so far, allyl etherification of guar gum is little reported in literature [16] .The objective of the present work was to synthesize allyl guar gum and the prepared samples were further characterized by Fourier transform Infra red spectroscopy (FT-IR), Thermogravimetric analysis (TGA) and Scanning electron microscope(SEM). .

EXPERIMENTAL

Materials and reagents

The Guar Gum with a moisture content of 8.5% and its viscosity of 1% Guar gum solution at 250C is 5000Cps and a particle size of 500µ was supplied by Dabur India Ltd , and Allyl Chloride was purchased from fisher Scientific , Isopropyl alcohol and tetramethyl ammonium chloride ,was purchased from Qualigens Fine Chemicals all reagents in the experiment were of pure grade .

General Procedure for Allyl Modified Guar Gum (AGG) :-

The Process involves the reaction of guar gum by its reaction with an allyl chloride in the presense of water –isopropanol solvent mixture to effect etherification **.** For this the guar- gum (GG) flour solution was dispersed to form a isopropyl solution in a three- necked clean round bottom flask 1 litre maintained at 40 0 C and purged with nitrogen after 1 h a predetermined ^{amount} of 50 %(w/v) aq solution of sodium hydroxide and a quaternary ammonium salt (phase transfer agent) tetramethyl ammonium chloride was added to the slurry and the mixture was stirred and heated to a temperature of 50o C. The reaction was initiated by dropwise addition of allyl chloride over a period of 30 min . The mixture was stirred vigorously for 6Hrs at 50 0C (Table 1). The reaction mixture was cooled gradually ^{dispersed} in acetone and the excess of alkali is neultalized with glacial acetic acid bringing the pH to 7 . The product than finally washed with acetone and then dried under vaccum.

Characterization of Allyl Guar- gum

FTIR -The guar gum and modified guar gum were analyzed by a Nicolet 380 Attenuated Total Reflactance –Fourier Transform Infrared (ATR-FTIR) spectrometry in transmittance mode .and samples was investigated with KBr pellet in absorbance mode .

Thermogravimetric analysis (TGA)- was used to conduct the thermal degradations under nitrogen atmosphere at TA instruments at a heating rate of 100 C/ min from ambient to 600 0C .

Scanning Electron Microscopy (SEM)- SEM Micrographs were taken on a Hitachi S-3700 microscope (japan) . The guar gum and allyl guar gum was sputterd with gold , observed and photographed .

RESULTS AND DISCUSSION

FTIR Analysis

The FT-IR spectrum of Allyl guar gum (AGG1) band located at 3418 cm-1,due to OH stretching indicating that some OH groups were allylated shown in Fig -2 . An absoprption peak appears at 1650 cm-1 in the infrared spectrum of allyl guar gum corresponding to the stretching vibrartion of C=C it demonstrate the existence of allyl groups respectively which may be attributed to the incorporation of allyl groups into the guar gum molecule [17].

Thermal analysis

The TGA curve of Guar gum and Allyl guar gum are shown in Fig -3 .The thermal stablity of the Guar gum is partially due to the hydrogen bonding occurring in the sample allylation reduces the hydrogen bonding thus could be the cause of the reduced thermal stability of the AGG derivatives. Thus it could be seen that allylation of guar gum resulted in AGG derivatives which have a terminal unsaturation group into the chain. The initial weight loss is due to presence of small amount of moisture in the sample. The second zone i.e degradation zone is starts at 240 oC . where the polymer decomposition takes place the rate of ^{weight} loss is increased upon increasing the temperature upto 340 0C nearly 75 % guar gum degrades below 400 0 C. A 16.2 % ^{residue} was obtained at 600 0C.In case of allyl guar gum AGG1 the polymer degradation starts at 224 0C.The polymer decomposition temperature indicated that allyl guar gum AGG1 lowers the initial decomposition temperature about 56.4% weight loss occurred between 200 and 400o C and 21.12% residue was obtained at 600^{0}C.

Scanning Electron Microscopy (SEM)

Surface morphological analysis of Guar gum and modified guar gum were confirmed by scanning electron microscope (SEM) and is presented in Fig 4. The size and appearance of the granules from allyl guar gum are rougher and more porous than that of Guar gum .

Conclusions -Allyl modified guar gum was efficiently prepared under moderate reaction conditions from the charcterization studies , It is being confirmed that the guar gum has been derivatised with allyl moiety resulting in a formation of a modified naturally occurring polysaccharide allyl Guar –gum .

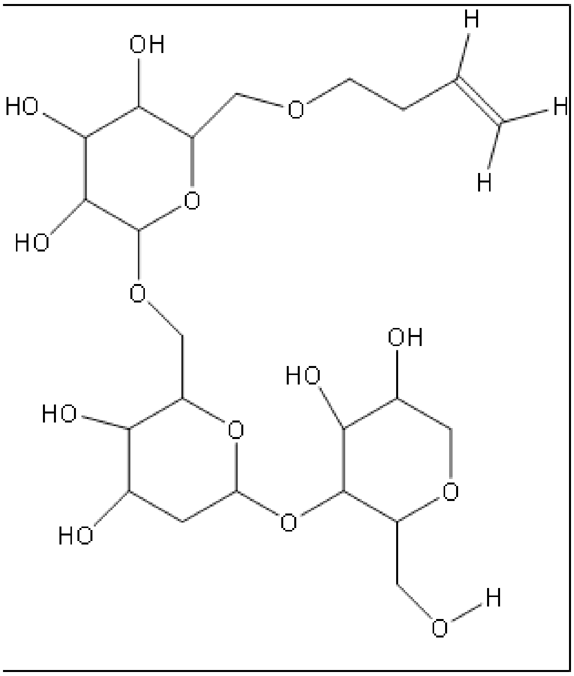

Fig1 – Chemical Structure of Allyl Guar Gum

Sample Name	Volume(ml) (NaoH)	Wt (gms) (Allylchloride)	Temperature ^{0} C	Time (Hrs)
AGG1	7	6.846	50	6

Table 1- Properties of Allylated guar gum

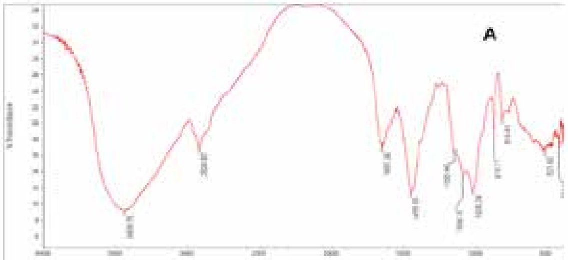

Fig 2. FTIR spectrum of Allyl guar gum (AGG1)

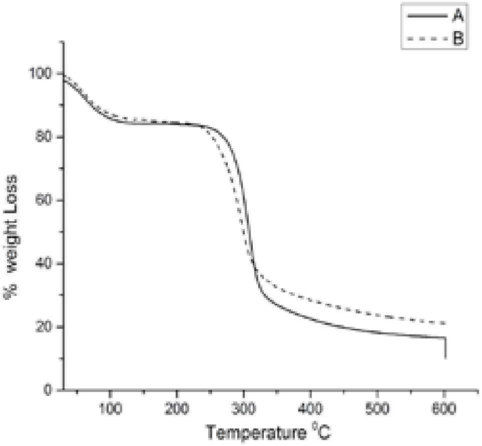

Fig3 – TGA curves of A =Guar gum and B= allyl guar gum

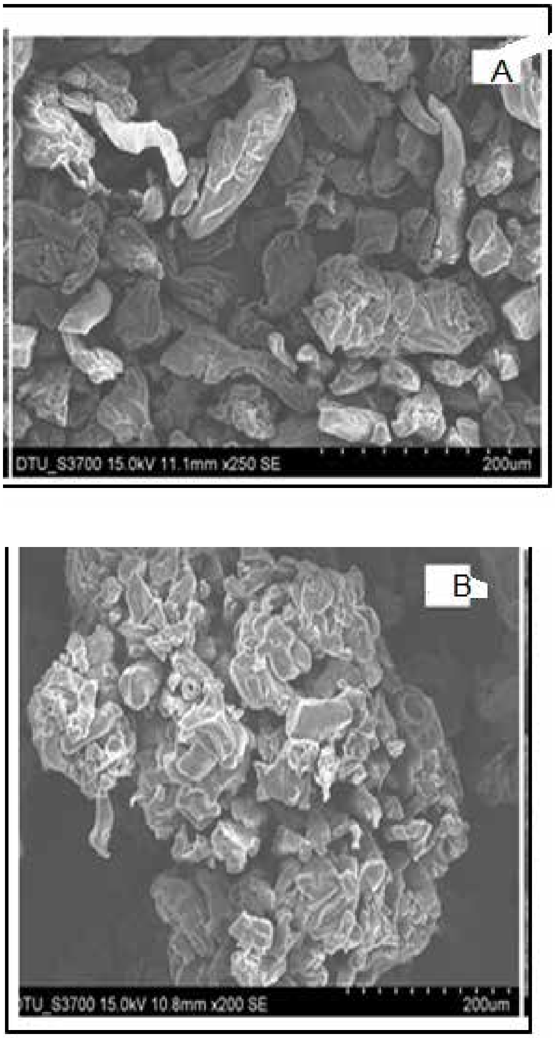

Fig 4-.Scanning electron Micrographs of A =Guar gum at X250 and C =allyl guar gumAGG1 at X200

REFERENCE

1. PrabhanjanH , Gharia M M & Srivastava H C ,Carbohydrate Polymers 11 279,(1989) | 2. Schneider R , and Sostar S , Dyes and Pigments , 57 , 7 , (2003) | 3. D. Risica, A. Barbetta, L. Vischetti, C. Cametti and M. Dentini, Polymer, 51, 1972 (2010) | 4. Funami T , . Kataoka Y , Omoto T , Yasunori G , Asai I and Nishinari K ,Food Hydrocolloids , 19(1),2005 | 5. Maurin V and Beauquey B, US Patent 6383993 (2002) | 6. Soppimath K S , Kukarni A R and T Aminabhavi ,J Controlled Release , 75 , 331, (2001) | 7. Gupta A.P and Arora Gopal Journal of Material Science and Engineering B1 28-33,(2011) | 8. Gupta A.P and Arora Gopal Der Chemica sinica ,3(5)1191-1197,(2012) | 9. Mothe C G , Correia D , de Franca F P ,. Journal of Thermal Analysis and Calorimetry85,31, (2006) | 10. ThimmaReddy T ,Tammishetti S, ,Polymer Degradation and stability 86 455-459(2004) . | 11. Godi G . Hritcu D and Popa M. Cellulose Chemistry & Technology , 45 (3-4) ,171-176 , 2011 | 12. D'Melo and Shenoy M A , ,Polymer Bulletin 61 ,235,(2008) | 13. Shenoy M and D.J. D'Melo, , e-Polymers 111, (2007) | 14. Xiao Wenfa ; Dong Litao , , International Conference on Mechanic Automation and Control Engineering (MACE) July 2011 | 15. Risica Daniela , Dentini Mariella , Vittorio Crescenzi Polymer 46 12247, (2005) | 16. .De Guia, United states Patent 4, 169,945 , (1979) |