

Composite of Methylmeth Acrylate –g – Guar Gum with Chitosan using Persulphate/Ascorbic Acid Redox Pair: Synthesis and Characterization

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Abstract: A novel composite of Guar Gum and Chitosan was prepared by the grafting of guar gum with methylmeth acrylate(MMA). Grafting process was initiated by using a redox pair of potassium persulphate and ascorbic acid that starts the crosslinking process between guar gum and MMA. Composite was made with this grafted guar gum and chitosan by condensation process and by thermal treatment. The synthesized composite was characterized by FT-IR, MASS, NMR, TGA, SEM, EDX analysis.

Keywords: redox pair, condensation, grafting, TGA, SEM analysis.

1 Introduction

Grafting is a process of modification in natural gums. Natural gums are present in nature in large amount and by grafting their physical and chemical properties changes for useful purpose. New semi synthetic polymers are formed by grafting with incorporating additional useful properties.¹

Guar gum (*Cyamopsis tetragonolobous*) is annual leguminous plant. Gum is found in the endosperm of seed of guar gum. Guar gum is high molecular weight polymer. Its chemical structure have (1→4)-β-D-manno-pyranosyl units with α-D-galacto-pyranosyl units attached by 1→6 linkages^{2,3} (fig. 1).

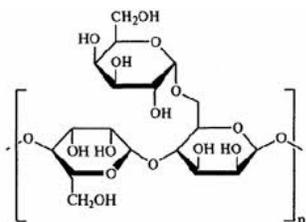


Fig. 1 Structure of Guar Gum

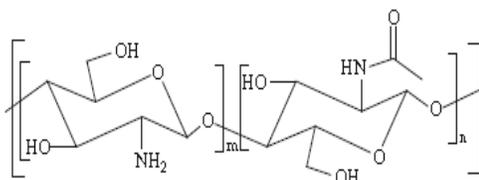


Fig. 2 Structure of Chitosan

Methylmeth acrylate was used for grafting purpose because it is capable to induce new bone growth. It easily enables the bond to transfer from material to newly grown bone. It is biodegradable material and gradually degrades⁴. Chitosan is a copolymer of N-acetyl glucosamine and glucosamine units. It is a homo polymer⁵. Chitosan is a linear polysaccharide composed of randomly distributed beta (1->4)-linked D-glucosamine (deacetylated unit) and N-acetyl-D-glucosamine (acetylated unit)⁶ (Fig.2).

Polymerization condensation method was selected among all the grafting methods⁷ because of high grafting efficiency, lesser cost and reaction in controlled manner⁸.

2 Materials and Method

2.1 Materials

Guar gum (Shri Ram Gum Industries Jodhpur), Methyl methacrylate(Merck), ascorbic acid, potassium persulphate(ases chemicals, jodhpur) used without further purification. Chitosan, Ethanol, Methanol, sodium hydroxide (1%), ammonium hydroxide (5N) were used.

2.2 Method

2.2.1 Synthesis of Methyl Methacrylate(MMA)-g-Guar gum

For the grafting process, guar gum(2gm), equal amount of ascorbic acid and MMA were added in 250ml of distil water. The mixture was heated at 35°C for 30 minutes and potassium persulphate and ascorbic acid redox pair was added to initiate the grafting process. Allowed this mixture to rest for one hour so that maximum grafting occurred. Pour that mixture into large quantity of Dimethyl formamide(DMF) to separate MMA-g- guar gum from the solution and wash it with DMF⁹. Percentage of grafting also calculated.

$$\% \text{ Grafting} = \frac{W_1 - W_0}{W_0} * 100$$

Here W_0 denoted weight of original Guar gum and W_1 denoted weight of grafted guar gum.

2.2.2 Saponification of MMA-g- Guar gum

The grafted guar gum was saponified with 1% solution of sodium hydroxide and heated to the boiling for for one hour. This saponified guar gum separated with the help of methanol and wash with ethanol.¹⁰

2.2.3 Preparation of chitosan composite with MMA-g-Guar gum

Saponified guar gums dissolved in distil water. Add 10 ml ethanol. In another beaker Chitosan dissolved in 5N ammonium hydroxide. Mix both the mixture and stirred at room temperature for 18-20 hours. Wash the mixture with distil water and evaporate excess liquid by heating at different range of temperature from 20°C to 100°C. Prepared composite were dried.

Scheme of composite formation:-

3.2 TGA analysis

TGA of the composite was carried out using TGA Q 500 V 6.7 Build 203 (Fig. 4). Under the inert atmospheric conditions thermal stability of compound was checked from room temperature to 600°C. There are three major breakdown was found in thermogram. The minor breakdown was occurring at 75°C and 4% weight loss occurs due to mainly evaporation of moisture. First major breakdown occurs at 230°C. At this point 14% weight loss done. Second breakdown was at 350°C and here almost 63% weight loss happened. This was because of breaking of polymer chain. The third major break in thermogram was found at 425°C and here 85% of total weight lost. After this when temperature increases there was no such major loss in weight found. This shows that the composite is thermally stable till around 425 °C temperatures that is much more then the pure guar or chitosan thermal stability.

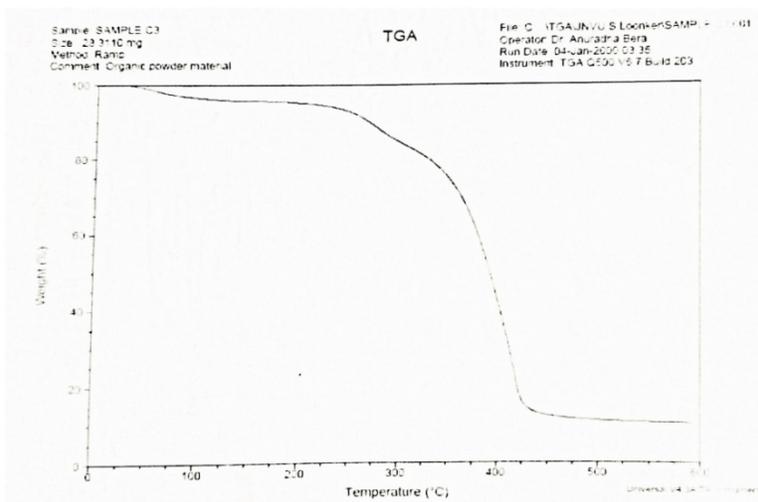


Fig.4 TGA thermogram

3.3 Mass analysis

Mass spectra was recorded on a JEOL-accuTOF JMS-T100LC mass spectrometer having a DART source. (fig.5) Dry Helium was used with 4 LPM flow rate for ionization at 350°C. The M+ value of the film is 879.15.

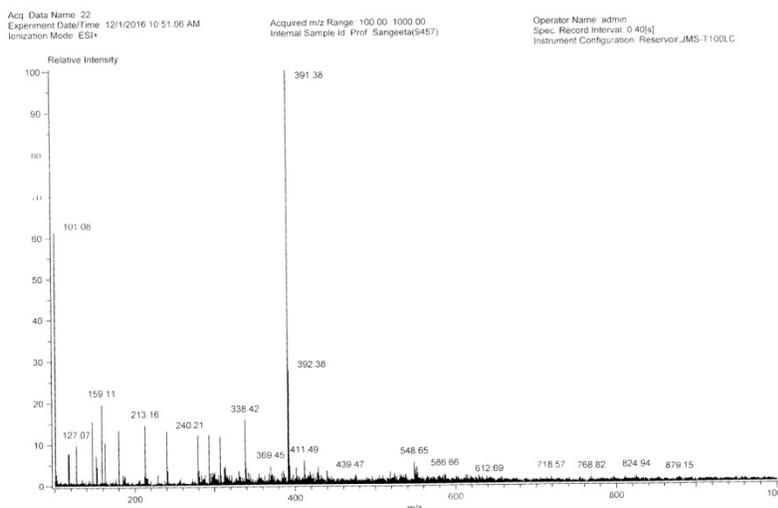


Fig. 5 MASS Spectra

3.4 ¹H NMR analysis

For the study of the newly synthesized composite ¹H NMR spectra (fig. 6) was taken by Bruker avll HD-300MHz FT-NMR. Chemical shift of different protons are different due to their chemical environment. The peak found at 7.9 ppm was due to protons of aromatic ring. Peak 3.3-3.5 due to methylene protons of –CH₂–OR group. Peak at 1.7ppm peak was of protons of amine group and 0.7-1.0 ppm peak was of protons of methyl group.

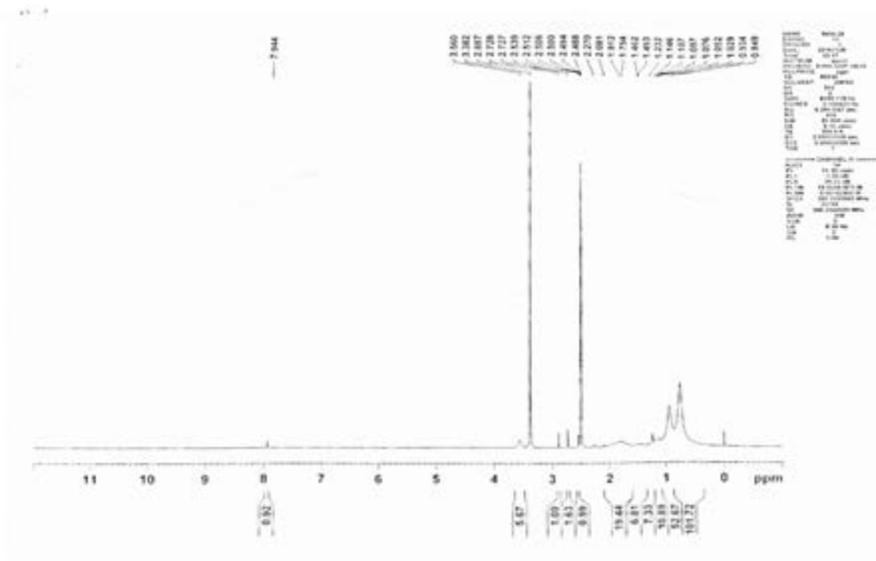


Fig. 6 ¹H NMR spectra

3.5 Scanning Electron Microscopy (SEM) and Elemental Analysis (EDX)

SEM analysis of the composite was done with JEOL JSM-7500F instrument. From the scanning electron image (fig.7), we can clearly analyze the surface of composite. Particles are overall uniform in size and spread over whole surface area.

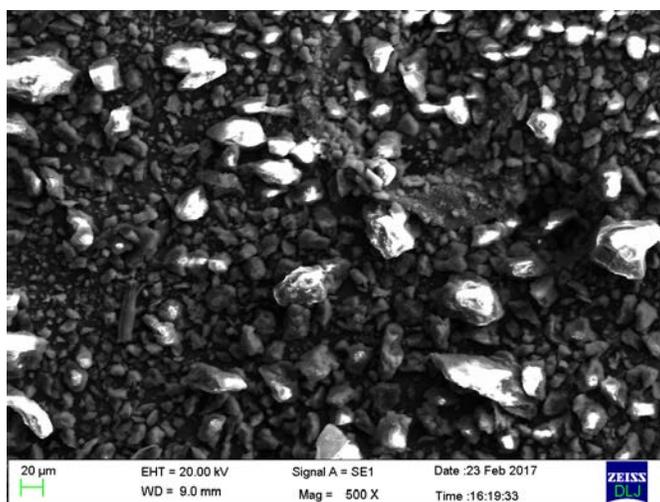


Fig. 7 SEM image

Elemental analysis is a process where a sample of some material is analyzed for its elemental composition such as carbon, hydrogen, nitrogen and sulphur. Elemental analysis of the composite was done and found the major percentage of carbon and oxygen. Composition of newly synthesized film is given in table 1.

Element	Weight %	Atomic %
Carbon	62.33	68.69
Oxygen	37.67	31.21

Table 1 EDX

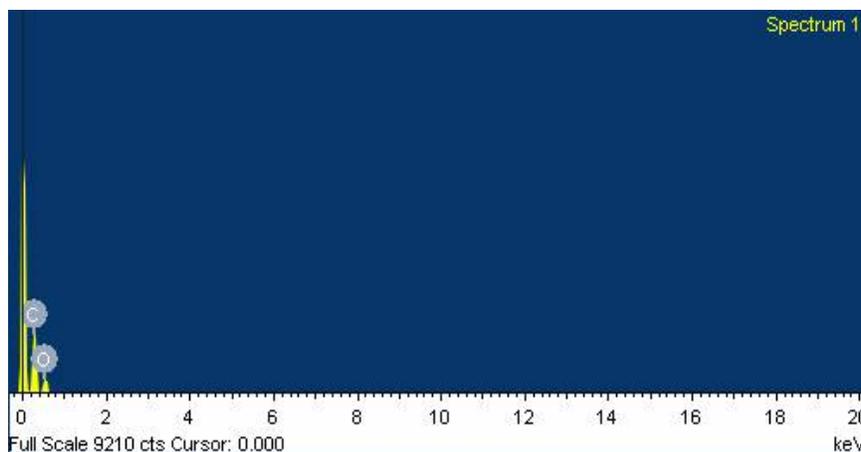


Fig. 8 EDX spectrum

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