Recovery and Synthesis of Guar Gum and its Derivatives

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Abstract: Despite the vast importance of green chemistry synthesis and characterization of natural biopolymers is need of time which eliminates the danger to health and environment. Polysaccharides are widely spread biopolymers with diversity of structure and properties. They are potential substitutes of petroleumderived synthetic products because they are easily available, nontoxic, biodegradable and biocompatible. Due to their fascinating properties they are used as a rheological modifier in food, pharmaceutical, paper, textile, oil and drilling and score of other industrial and commercial sectors. Modification of hydrophilic backbone of these biopolymers diversifies and enhances its applications and functionality. Guar gum is an outstanding representative of green, ecofriendaly biopolymers This Review deals with structure, properties and potential uses of green biopolymer guar gum. Different methodologies to modify guar to enhance and diversify its properties and application range are discussed and reviewed. There is a high year-to-year variation in production of guar, and consequently, in exports of guar and its derivatives. Guar gum is mainly used in the food and bakery industry, the food safety concerns are becoming important for the guar processing industry. The preparedness of guar split and guar gum manufacturing industries for these food safety concerns, high fluctuations in area, production and productivity of guar seed, high volatile prices of guar seed and gum splits, are crucial limitations to the growth of guar industry.

Keywords: Biopolymers, Guar gum, Green, Modifications, and Hydrophilic backbone.

1. INTRODUCTION

Despite the vast importance of green chemistry synthesis and characterization of natural biopolymers is need of time which eliminates the danger to health and environment. Polysaccharides are widely spread biopolymers with diversity of structure and properties. They are potential substitutes of petroleum-derived synthetic products because they are easily available, nontoxic, biodegradable and biocompatible. Due to their fascinating properties they are used as a rheological modifier in food, pharmaceutical, paper, textile, oil and drilling and score of other industrial and commercial sectors. Modification of hydrophilic backbone of these biopolymers diversifies and enhances its applications and functionality. Guar gum is an outstanding representative of green, eco-friendly biopolymers. This deals with structure, properties and potential uses of green biopolymer guar gum. Different methodologies to modify guar to enhance and diversify its properties and application.

Guar gum (GG) is an edible carbohydrate polymer which is useful as a thickening agent for water and as a reagent for absorption and hydrogen bonding with mineral and cellulosic surfaces. GG is a galactomannan. It consists of a straight chain of mannose units joined by β —D (1→4) linkages having α —D galactopyranose units attached to this linear chain by (1→6) linkages. The molecular weight of GG is about 220,000. GG is being used in explosives, foods, cosmetics and pharmaceuticals, and in mining, paper and textile industries, mostly as a water binder. Guar gum is produced from guar seeds. Guar seed is dicotyledonous, having a diameter of approximately 8 mm. It consists of 14-17% hull, 43-47% germ and 35-42% endosperm.

1.1. Literature Review

Due to its unique rheology modifying properties, it is being widely used across a broad spectrum of industries viz. oil well drilling, textile, paper, paint, cement, cosmetic, food, pharmaceutical etc. India is the major producer of guar in the world and its contribution to the world- production is around 80%. In India, Rajasthan and Haryana States contribute 85% of the total production of India. India is a major exporter of guar gum.

1.1.1. Guar Plant

Guar also known as cluster bean. Guar is being grown for seed, green fodder, vegetable and green manuring. It is an annual plant, about 4 feet high, vertically stalked, with large leaves and clusters of pods (Fig. 1). Each pod is about 5-8 cm long and has on an average 6-9 small grayish-white pea shaped seeds (Fig. 2A & 2B). The pods are used as a green vegetable or as a cattle feed besides the industrial extraction of guar gum. Guar grows best in sandy soils. It needs moderate, intermittent rainfall with lots of sunshine. The crop is sown after the first rains in July and harvested in October-November. It is a short duration crop and is harvested within 3-4 months of its plantation. Guar is a rain dependent crop; rainfall influences the yield of the crop.



Fig1. Guar plant

1.1.2. Guar Gum

Guar gum, also called guaran, is a natural accumulated hydrocolloid stored in the endosperm of the seeds of the guar plant. From the chemical point of view, guar is a gallactomannan with a characteristically configured macromolecular structure. The macromolecular structure of guar lies between a spherocolloid (like amylopectin) and a linear hydrocolloid (like cellulose). The backbone of the carbohydrate is made of a chain of (1 to <4) glycosidic linked mannose units, on which every second unit is branched with a (1 to <6) linked gallactose. The chemical structure is shown in Figure 3. Like most polysaccharides, guar has two or three free hydroxyl groups on the mannose unit of the main chain or the gallactose side chains. These are available for bonding and can be utilized in bonding with the numerous hydroxyl groups. It is a white to creamish amorphous powder, nearly odourless, dispersible in cold and hot water to form a translucent and colorless to whitish colloidal solution. Guar gum is an emulsifier, thickener, and stabilizer approved for use in a wide range of foods, cosmetics, and pharmaceuticals.

1.1.3. Chemical Structure of Guar Gum

GG is a galactomannan. It consists of a straight chain of mannose units joined by β —D (1 \rightarrow 4) linkages having α —D galactopyranose units attached to this linear chain by (1 \rightarrow 6) linkages.



Fig2. Chemical structure of guar gum

The ratio of mannose to galactose unit (M/G) ranges from 1.8:1 to 2:1 due to variation in geographical 9 origins. Firstly, it believes that galactose side groups are more uniformly distributed at regular intervals along the mannose backbone.

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1.1.4. Constituents of Guar Seed

The guar seeds are dicotyledonous containing two endosperm halves per seed. The endosperm accounts for about one third of the bean weight and contains the majority of wonderful Galactomannan (gum). The remaining two thirds are the hull and germ which are very high in protein and fiber.



Fig3. Guar seed



Fig4. (a) Guar seeds (b) guar endosperm

Table1. Constituents of guar seed

Part Of Seed	Protein %	Ether Extract%	Ash %	Moisture %	Fibres %	Type Of Sugar
Hull (14-17%)	5	0.3	4	10	36	D-Glucose
Endosperm (35-42%)	5	0.6	0.6	10	1.5	Galactomannon
Germ (43-47%)	55.3	5.2	4.6	10	18	Glucose



1.1.5. Properties of Guar Gum

The main properties of Guar Gum are

- It is soluble in hot & cold water but insoluble in most organic solvents.
- It has strong hydrogen bonding properties.
- It has excellent thickening, Emulsion, Stabilizing and film forming properties.
- At very low concentration, Guar Gum has excellent settling (Flocculation) properties and it acts as a filter aid.
- It is non ionic and maintains a constant high viscosity over a broad range of ph.
- It is compatible with a variety of inorganic and organic substances including certain dyes and various constituents of food.
- The viscosity of Guar Gum solution increase gradually with increasing concentration of Guar Gum in water.
- The viscosity of Guar Gum is influenced by temperature, ph, presence of salts and other solids.
- It has excellent ability to control rheology by economic water phase management.

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1.1.6. Application

- Guar gum and its derivatives are widely used in various industries as per its needs. It
- is used in industries such as food, textile, pharmaceuticals, personal care, health
- Care, nutrition, cosmetics, paper, explosives, mining and oil well drilling. Guar gum
- mainly functions as a thickener, emulsifier, stabilizer, binding agent, gelling agent,
- Natural fiber, flocculant, fracturing agent etc.

2. METHODS

2.1. Manufacturing Process of Guar Gum

Manufacturing process of guar gum is, Guar splits are obtained after separation of the husk and the germ. After heat treatment, the hull is easy to separate by either attrition milling or various types of impact mills. The endosperm is recovered by sieving from the finer germ and hull fractions, and then milled to obtain powdered guar gum. The guar gum may be further clarified (purified) by dissolution in water, precipitation and recovery with ethanol or isopropanol.

2.2. Recovery of Guar Gum Powder from Guar Seeds

Prepared sample of guar gum powder from guar seeds by using simple process. Guar seeds heated in double or more amount of water to boil it. Then attrition process is carried out to separate endosperm from husk and germ. Then dried endosperm is passed through size reduction equipment. Then sieving process is carried out to separate different particle size.



Fig6. *Manufacturing of guar gum powder*

Stepwise procedure for manufacturing of guar gum powder from guar seed are as given below



Fig7. Guar seeds



Fig8. Heating of guar seeds



Fig9. Endosperms



Fig10. Germ & husk



Fig11. Guar gum powder

2.3. Manufacturing of Adhesive from Guar Gum Powder

In this process 1gm (w/v) guar gum powder is blended with water for some time. In this process blending is most important for manufacturing of adhesive. We can add upto1-5% guar gum powder in water, as the concentration increases the viscosity also increased.

2.4. Manufacturing Process of Guar Gum Derivatives

2.4.1. Modification by Chemical Reactions

A) Carboxymethylation of guar gum

PROCESS

- A 70 gm sample of guar gum was swelled in 400 ml of 2-propanol with stirring.
- Then 24.8gm of NaOH was added over a period of 20 min. & the mixture was allowed to stand for further swelling for 60 min. at room temp.
- Then a 60 gm of SCA (sodium chloroacetate) was added to the mixture over a period of 30 min. and the mixture was allowed to react for 1 hour.
- The temperature of the reaction was then raised to 70°c within 1 hour. The reaction was then allowed to proceed for 2-3 hour.
- Then pale yellow color product was isolated by filtration.
- Washed with methanol and dried at room temperature for 2 days.

B) Cross linked guar gum

PROCESS

- 4 gm of guar gum was dispersed for 1 hr. at room temperature in 400 ml of distilled water with the help of mechanical stirrer.
- The dispersion was kept aside for 2 hrs. For swelling.

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- Then 0.8 gm of borax was added in 80 ml of water. This mixture was added to the guar gum dispersion & stirred for 30 min. with the help of mechanical stirrer.
- Then kept aside for another 4 hrs. without stirring.
- The formed hydrogel was rinsed with distilled water to remove untreated borax and then the hydrogel was dried at room temperature for 3 days.
- The dried borax cross linked guar gum passed through size reduction equipment.

2.4.2. Modification by Mechanical Process

A) Normal guar gum:

Manufacturing process of Normal guar gum:- In this process guar splits are manufacturing using conventional methods & finally converted into fine 200 mesh powder.

B) Slow hydrating guar gum:

Manufacturing process of Slow hydrating guar gum:- In this process guar splits are grind in coarse particle size with reduced prehydration and sieving through higher pore sieves so as to achieve coarser powder with dehydrating properties.

C) Fast hydrating guar gum:

Manufacturing process of fast hydrating guar gum:- In this process over swelled prehydrated guar splits are grind in very fine particle size so as to achieve fast hydrating properties.

2.5. Manufacturing Process of Guar Gum and Water Soluble Polymer Blend Film

Solution blends were prepared using the required volumes of guar gum and water soluble polymer solutions. These blends were mixed thoroughly to result in a uniform homogeneous solution. The films were prepared by casting onto a sheet mold and evaporating the water at room temperature in an atmosphere.

3. RESULTS AND DISCUSSION

Characterization of physical and chemical properties of guar gum and guar gum derivatives. The sample has been characterized by different test. FTIR, Thermo gravimetric Analysis.

3.1. FT-IR (Fourier Transform Infrared Spectrometer)

Infrared (IR) spectroscopy looks at the vibrational motion of bonds in molecules. By identifying the types of bonds present in a certain compound, one is able to gain insight into that compound's structure. There are certain characteristics of bonds that are dependent upon the compound's complete structure, namely the wavelength and amount of energy absorbed. What the infrared spectrometer does is give us an understanding of the structure and bonding present in a molecule by looking at the vibrational motions of which it undergoes. It is through these means that a compound is able to be identified since every compound has a unique spectrum (Silberberg 343). It should be noted, however, that infrared spectroscopy will only work for compounds that have a dipole moment (Skoog 382).

The peaks on the IR spectrum correspond to the energies absorbed by the various functional groups. By noting the functional groups in a molecule, interpretation of an IR spectrum can indicate the identity of a molecule by the presence or absence of such peaks. The fingerprint region (1400 - 990 cm-1) can also help identify a compound since this region consists of peaks that are attributed to the molecule as whole and not just specific functional groups (Zubrick 271). It is also possible, through analyzing and comparing IR spectra, to observe changes in a compound by looking for shifts in band frequency or the appearance of new features in the spectra (Mitra 38-39).

The FTIR-ATR spectrum of the pure guar gum film shows absorption band at around 3280.76cm⁻¹ for the O—H stretching vibrations and another band at 2940.52 cm⁻¹ assigned as C—H stretching vibration band, for the in-plane bending of O—H group at around 1423.55 cm⁻¹, strong band at 1249.98cm⁻¹ C—O stretching vibration of alcoholic group present in guar gum.

Table2. Characteristic IR Absorptions

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Frequency, cm ¹	Bond	Functional group		
3640–3610 (s, sh)	O–H stretch, free hydroxyl	alcohols, phenols		
3500-3200 (s,b)	O–H stretch, H–bonded	alcohols, phenols		
3400-3250 (m)	N–H stretch	1°,2° amines, amides		
3300–2500 (m)	O–H stretch	carboxylic acids		
3330–3270 (n, s)	–C≡C–H:C–H stretch	alkynes (terminal)		
3100–3000 (s)	C–H stretch	aromatics		
3100–3000 (m)	=C–H stretch	alkenes		
3000–2850 (m)	C–H stretch	alkenes		
2830–2695 (m)	H–C=O:C–H stretch	aldehydes		
2260–2210 (v)	C≡N stretch	nitriles		
2260–2100(w)	–C≡C– stretch	alkenes		
1760–1665 (s)	C=O stretch	carbonyls (general)		
1760–1690 (s)	C=O stretch	carboxylic acids		
1750–1735 (s)	C=O stretch	esters, saturated aliphatic		
1740–1720 (s)	C=O stretch	aldehydes, saturated aliphatic		
1730–1715 (s)	C=O stretch	A, βunsaturatedesters		
1715 (s)	C=O stretch	ketones, saturated aliphatic		
1710–1665 (s)	C=O stretch	A,β–unsaturated aldehydes, ketones		
1680–1640 (m)	-C=C- stretch	alkenes		
1650–1580 (m)	N–H bend	1° amines		
1600–1585 (m)	C–C stretch (in–ring)	aromatics		
1550–1475 (s)	N–O asymmetric stretch	nitro compounds		
1500–1400 (m)	C–C stretch (in–ring)	aromatics		
1470–1450 (m)	C–H bend	alkanes		
1370–1350 (m)	C–H rock	alkanes		
1360–1290 (m)	N–O symmetric stretch	nitro compounds		
1335–1250 (s)	C–N stretch	aromatic amines		
1320–1000 (s)	C–O stretch	alcohols, carboxylic acids, esters, ether		
1300–1150 (m)	$C-Hwag(-CH_2X)$	alkyl halides		
1250–1020 (m)	C–N stretch	aliphatic amines		
1000–650 (s)	=C–H bend	alkenes		
950–910 (m)	O–H bend	carboxylic acids		
910–665 (s, b)	N–H wag	1°, 2° amines		
900–675 (s)	С–Н "оор"	aromatics		
850–550 (m)	C–Cl stretch	alkyl halides		
725–720 (m)	C–H rock	alkanes		
700–610 (b, s)	$-C \equiv C - H$: C-H bend	alkynes		
690–515 (m)	C–Br stretch	alkyl halides		

m=*medium*, *w*=*weak*, *s*=*strong*, *n*=*narrow*, *b*=*broad*, *s*h=*sharp*



Fig12. FTIR- spectrum of GG

3.2. Thermo-Gravimetric Analysis

Thermal degradation determined by Thermo-gravimetric analysis. The glass transition temperature of a polymer network is affected by the cross-linking density as well as the chemical structure; increased aromatic content should result in higher Tg where as reduced cross-linking density would have the opposite effect.

4. CONCLUSION

Guar gum is versed with multiple physical and chemical properties which make it a strong candidate in the world of excipients in food, cosmetic and pharmaceuticals. Guar gum and its derivatives are directly used in therapy in some of the disorders. The chemistry of guar gum allows it to be chemically modified to obtain derivatives of desirable properties which are less expensive, biodegradable and eco-friendly.

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