



# Radiation dose dependent change in physicochemical, mechanical and barrier properties of guar gum based films



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## ABSTRACT

Mechanical and water vapor barrier properties of biodegradable films prepared from radiation processed guar gum were investigated. Films prepared from GG irradiated up to 500 Gy demonstrated significantly higher tensile strength as compared to non-irradiated control films. This improvement in tensile strength observed was demonstrated to be due to the ordering of polymer structures as confirmed by small angle X-ray scattering analysis. Exposure to doses higher than 500 Gy, however, resulted in a dose dependent decrease in tensile strength. A dose dependent decrease in puncture strength with no significant differences in the percent elongation was also observed at all the doses studied. Water vapor barrier properties of films improved up to 15% due to radiation processing. Radiation processing at lower doses for improving mechanical and barrier properties of guar based packaging films is demonstrated here for the first time.

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## 1. Introduction

In the past twenty years, the production and use of plastics in the world has increased enormously to about 200 million tons per year. Packaging constitutes the largest market for plastics, amounting to over 12 million tons per year (Rhim and Perry, 2007). Increasing demand for synthetic packaging materials has put tremendous pressure on the environment because of their poor biodegradability and non-renewability (Ghasemlou, Khodaiyan, Oromiehie, & Yarmand, 2011). This has led to a search for packaging materials that are biodegradable as well as recyclable (Mangiaccapra, Gorrasi, Sorrentino, & Vittoria, 2006). One of the alternatives is the development of packaging material from biopolymers (i.e. protein, polysaccharide and lipid) that are biodegradable, non-toxic and derived from completely renewable resources. Among the biopolymers, polysaccharides are the most widely used for preparation of packaging films.

Widely studied polysaccharides for edible or biodegradable films are: starch, chitosan, carrageenan, and galactomannans. In packaging industry galactomannan is used as edible coating

because it forms very thick aqueous solution at low concentration (Cerqueira et al., 2011), is an excellent emulsifier and non-toxic (Cerqueira, Lima, Teixeira, Moreira, & Vicente, 2009). Guar gum (GG) is a type of galactomannan, derived from endosperm of an annual legume plant *Cyamopsis tetragonoloba*. India accounts for 80 percent of world production of GG. It is a heteropolysaccharide of a mannose (i.e. (1-4)-linked  $\beta$ -D-mannopyranose) backbone with galactose side groups ((1-6)-linked  $\alpha$ -D-galactopyranose) (Aydinli, Tutas, & Bozdemir, 2004; Das, Ara, Dutta, & Mukherjee, 2011; Martins, Cerqueira, Souza, Carmo, & Vicente, 2010). It is mainly used in paper, food and pharmaceutical industries (Chudzikowski, 1971).

Major limitations in the use of biopolymers as packaging materials are their relatively poor mechanical and barrier properties such as tensile strength and water vapor transmission rate as compared to their non-biodegradable counterparts (Cha and Chinnan, 2004; Kang and Min, 2010; Petersson and Oksman, 2006). This has resulted in a greater focus on improving the properties of these polymers to match the commercially available packaging material. Various chemical and physical methods have been used for improving biopolymer film properties. Among the physical methods, addition of plasticizer for improving mechanical properties of biodegradable films has been extensively reported. This increases the percentage elongation of films by forming hydrogen bond with the polymer and reducing polymeric interactions. Polysaccharide based films are commonly plasticized with polyols such as glycerol (Garcia, Ribba, Dufresn, Aranguren, & Goyanes, 2011).

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Among the chemical methods, chemical modification of guar galactomannan with benzamide for preparation of water resistant films has been recently reported (Das et al., 2011). Mikkonen et al. (2007) used enzymatic depolymerization for improving mechanical properties of GG film. Gamma irradiation has been widely used for the improvement of mechanical properties of pectin (Kang, Jo, Lee, Kwon, & Byun, 2005), starch (Kim, Jo, Park, & Byun, 2008) and calcium caseinate edible films (Vachon et al., 2000). Use of gamma irradiation for GG depolymerization has been previously reported (Gupta, Shah, Sanyal, Variyar & Sharma, 2009). There are several advantages associated with gamma irradiation such as convenience, eco friendly nature of the process (Hwang, Jung, Kuk, Choi, & Nho, 2010) and short processing time.

Physical treatments such as gamma irradiation could possibly change conformations of polymers in solution. Several investigations have shown that the conformation and morphology of polymer chains affect the physical properties of the polymer. Polymer chain conformation and chain correlation can be estimated by small angle X-ray scattering (SAXS) (Winokur, Skotheim, Elsenbaumer, & Reynolds, 1998). SAXS arises from the fluctuations of electron density in a mesoscopic length scale (1–100 nm) in a specimen and hence scattering profile contains the information about the size/size distribution and shape of the inhomogeneities (Glatter and Kratky, 1982).

To the best of our knowledge, no reports exist till date on the assessment of the effects of gamma irradiation on mechanical and barrier properties of GG based films. The main objective of this work was to study the effect of irradiation on physicochemical properties of GG and to determine the impact of these properties on the mechanical and barrier properties of films prepared from the irradiated GG.

## 2. Materials and methods

### 2.1. Purification of GG

Purification of guar gum was carried out as per procedure detailed earlier by Jumel, Harding, and Mitchell (1996). In brief, 2.5 g of GG (Merck India Ltd.) was dissolved in 250 ml of distilled water by using shear mixer (Omni mixer, Sorvall, USA) at speed 2 for 2 min. Solution obtained was kept overnight on magnetic stirrer at room temperature ( $25 \pm 2^\circ\text{C}$ ). Resulting solution was centrifuged at 9000 rpm for 30 min for removal of insoluble impurities. Ethanol was added to supernatant in the proportion of 2:1 and resulting mixture was kept overnight for precipitation of GG. The precipitate obtained was freeze dried to obtain dry purified GG powder. Yield obtained by above purification procedure was 60%.

### 2.2. Irradiation of GG

Purified GG as dried powder was exposed to gamma radiation processing using a  $^{60}\text{Co}$  gamma irradiator having dose rate of 4.1 kGy/hr (GC-5000, BRIT, India) at room temperature. GG was subjected to a dose of (0.25, 0.5, 0.75, 1, 5, 10, 25 and 50 kGy). In addition, films prepared from control unirradiated GG were subjected to gamma radiation to a dose of (1, 5, 10, 25, 50 and 100 kGy) after 7 days of conditioning.

### 2.3. Viscosity average molecular weight analysis by Ostwald viscometer

Viscosity average molecular weight of GG post irradiation was measured using Ostwald's viscometer at constant temperature of  $24 \pm 1^\circ\text{C}$ . 0.1% w/v aqueous solution was prepared from control

and irradiated GG and specific viscosity ( $\eta_{sp}$ ) was obtained using following Eq. (1):

$$[\eta_{sp}] = \frac{(t - t_0)}{t_0} \quad (1)$$

where  $t$  = flow time of a polymer solution through viscometer;  $t_0$  = flow time of the pure solvent through the same viscometer.

Intrinsic viscosity ( $\eta$ ) was then calculated from  $\eta_{sp}$  using following Eq. (2):

$$[\eta] = \frac{[\eta_{sp}]}{c} \quad (2)$$

where  $c$  = polymer concentration.

Viscosity-average molecular weight ( $M_v$ ) was calculated from  $\eta$  (Eq. (3)) (Vega, Lima, & Pinto, 2001).

$$[\eta] = KM_v^a \quad (3)$$

where  $K$  and  $a$  are the parameters that depend on the solvent-polymer pair. The  $a$  and  $K$  values used for guar galactomannan were 0.72 and  $5.13 \times 10^{-4}$ , respectively (Beer, Wood, & Weisz, 1999).

### 2.4. Molecular weight and polydispersity index analysis by gel permeation chromatography

Number average molecular weight ( $M_n$ ), weight average molecular weight ( $M_w$ ) and polydispersity index (PDI) ( $M_w/M_n$ ) were determined by gel permeation chromatography (GPC) using a column (300 mm length  $\times$  4.6 mm I.D.), 5u Biobasic SEC-1000, Thermo Scientific, UK). The HPLC system (Ultimate 3000, Dionex Corporation, Germany) having differential refractive index detector (RI-101, Shodex Corporation, USA) was used. The mobile phase was deionized water (Millipore, Bedford, MA) and the flow rate was fixed at 0.6 mL/min. All GG samples were injected (20  $\mu\text{L}$ ) as their aqueous solutions at concentrations of 0.2% (w/v) which were centrifuged at 12,000 rpm for 15 min prior to analysis. The column was calibrated using pullulan standards (Fluka Analytical, St. Louis, USA) ranging from molecular weights of 6000 to 2,560,000 Da. Pullulan standards were analyzed using similar HPLC conditions described above.

Number average molecular weight ( $M_n$ ) was calculated by following Eq. (4):

$$M_n = \sum \left( \frac{N_i}{\sum N_i} \times M_i \right) \quad (4)$$

where  $N_i$  = detector response at a particular time

$\sum N_i$  = Total detector response

$M_i$  = Molecular weight at given time.

Weight average molecular weight was calculated by following Eq. (5):

$$M_w = \sum \left( \frac{A_i \times M_i}{\sum A_i} \right) \quad (5)$$

where  $A_i = N_i \times M_i$

Based on  $M_n$  and  $M_w$ , PDI was calculated using equation given below:

$$\text{PDI} = \frac{M_w}{M_n} \quad (6)$$

### 2.5. Determination of mannose to galactose ratio of GG by HPLC

Control and irradiated GG samples were hydrolyzed by 1N sulphuric acid at  $90^\circ\text{C}$  for 5 h. After hydrolysis samples were neutralized using barium hydroxide and barium sulfate precipitates

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