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Short Communication

# *Ipomoea turpethum* seeds: a potential source of commercial gum

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**Abstract**

A non-ionic water-soluble galactomannan, having a galactose and mannose in 1:2 molar ratio was isolated from endosperm of the seeds. Seed gum has a branched structure consisting of a linear chain of β (1 → 4) linked mannopyranosyl units with D-galactose side chains attached through α (1 → 6) linkage to the main chain, a fundamental structural pattern found in other seed galactomannans like guar, carob, locust bean, tara and dhaincha commercial gums. The gum was subjected to viscosity behavior study at different conditions of concentrations, temperature and pH. Gum solutions showed similar behavior to the guar and were found to be stable over a wide range of pH. With borate ions reversible cohesive gels resulted. Gel formation with transition metal ions is pH dependent and non-reversible. Thus, the seed gum from *Ipomoea turpethum* show similarity in structural pattern and properties to guar gum. Grafting of polyacrylamide on to the seed gum in aqueous medium, initiated by potassium persulphate/ascorbic acid redox system has been carried out at 35 ± 0.2 °C in the presence of atmospheric oxygen and $Ag^{+}$ ions. After grafting the viscosity of the gum solution was found to increase tremendously. *I. turpethum* seed gum thus in natural form and, after modification by grafting may find use as a commercial gum. 

*Keywords:* Commercial gum; *Ipomoea turpethum*; Potassium persulphate

## 1. Introduction

*Ipomoea turpethum* (N.O. Convolvulaceae) is a perennial plant (Kirtikar & Basu, 1975) with a much-twisted stem and is found through out India. It is reported to be highly medicinal and is cultivated occasionally in gardens as an ornamental plant. Some hydroxy fatty acids (Roos & Bakker, 1991), butulin, lupeol and sitosterol (Jain & Saxena, 1987) have been found in the plant. Seed gums are important vegetable products used in various industries (Davidson, 1980; Whistler, 1973) like paper, textile, cosmetics, food and pharmaceuticals. In view of the easy availability of the plant and the high demand for seed gums throughout the world, the present investigations have been carried out to determine if *I. turpethum* seeds can be used as a potential source of the commercial gum. Graft co-polymerization of guar gum (Bajpai, Alka, & Sandeep, 1990) has been reported to impart desirable properties so the *I. turpethum* seed gum was subjected to graft co-polymerization.

## 2. Materials and methods

The seeds were collected locally and identified at the *Botanical Survey of India*, Allahabad, India. Solutions were concentrated at diminished pressure at 60–62 °C. Paper chromatography was carried out at room temperature with solvent system (A) 1 butanol–ethanol–water (Hirst & Jones, 1949) (5:1:4); (B) 1 butanol–2 propanol–water (Rizvi, Gupta, & Kaul, 1971) (11:6:3); (C) ethyl acetate–pyridine–water (Aspinall, Begbie, & Mackay, 1962) (10:4:3); (D) water–pyridine–water (Meier, 1960) (2:1:2), with detection using aniline hydrogen phthalate. IR spectra were recorded by Perkin–Elmer model 457 infrared spectrophotometer. Acrylamide (E. Merck) was recrystallized twice from methanol (G.R.) and dried under vacuum. Ascorbic acid and potassium persulphate (B.D.H. Analar Grade) were used without further purification. All solutions were prepared from doubly distilled water.

### 2.1. Isolation of the seed gum

Dried crushed seeds were extracted successively with light petroleum and ethanol to defat and decolorize, respectively, then extracted with 1% aqueous acetic acid

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and extract was added slowly, with stirring to large excess of ethanol. The crude gum was collected, washed with ethanol and dried (yield 2.3 g/100 g).

### 2.2. Purification of the seed gum

The crude polysaccharide was purified through barium complexing by preparing 2.5% (w/v) solution of the gum by continuous stirring for 12 h at 60 °C and precipitating with saturated barium hydroxide solution. The complex was separated by centrifugation and taken in 1 M acetic acid, stirred for 8 h, centrifuged and precipitated with ethanol. It was washed with 70, 80, 90, 95% ethanol. The sample was finally purified by dialysis and filtration through various Millipore membranes. The pure seed gum was a non-reducing, white, amorphous material with ash content 0.28% and $[\alpha]_D^{25} = +58°$ (water).

### 2.3. Investigation of the structure of the polysaccharide

The pure seed gum was completely hydrolyzed with 1 M trifluoroacetic acid (4 h, at 100 °C). Paper chromatography (solvent-B) of the hydrolysate revealed the presence of galactose ($R_f$ 0.15) and mannose ($R_f$ 0.21). Identities and configurations of the monosaccharides were confirmed by co-chromatography with authentic samples and preparations of derivatives; D-galactose, mp 163 °C, $[\alpha]_D^{30} = +80°$ (water); D-galactose phenyl hydrazone, mp 153 °C; D-mannose, mp 131 °C, $[\alpha]_D^{30} = +14°$ (water); D-mannose phenyl hydrazone, mp 198 °C. The ratio of the constituent monosaccharides was determined by GLC (Kapoor, Chanzy, & Travel, 1995). The seed gum hydrolysate was concentrated by evaporation, the residue reduced with sodium borohydride and the products acetylated with pyridine–acetic anhydride (1:1 v/v, 1 h at 100 °C). The resulting alditol acetates were analyzed by GLC using a model Neukon 5700 Gas Chromatograph equipped with flame ionization detector, at 190° with a Superleco SP 2380 column ($3.0 \times 0.53$ mm$^2$), the carrier gas being nitrogen. The ratio of D-galactose to D-mannose was found to be 1.01:2.03. The polysaccharide was hydrolyzed (Smith & Montgomery, 1959) with 25 mM $H_2SO_4$ at 100 °C for 6 h. PC of the hydrolzate showed that galactose was liberated first followed by D-mannose. Metaperiodate oxidation studies (Singh, Mishra, Khare, & Gupta, 1997) revealed that 0.8200 mol of metaperiodate were consumed with the liberation of 0.1950 mol of formic acid per 100 g of the polysaccharide indicating 31.62% of the end groups.

The seed gum was first methylated by Haworth's method (Haworth, 1915) followed by Hakomori method (Hakomori, 1964) to yield a fully methylated product, $[\alpha]_D^{25} = +40°$ (Chloroform). The completely methylated seed gum having no absorption at 3600–3400 cm$^{-1}$ was boiled under reflux with 90% aqueous HCOOH for 6 h then with 1 M $H_2SO_4$ for 14 h at 100 °C. The products were fractionated on Whatman no 3MM paper (solvent-A) to give following methylated sugars. (I) 2,3,4,6-tetra-*O*-methyl-D-galactose, mp 72–73 °C, $[\alpha]_D^{32} = +120°$ (C 1, water) (cf. literature (Robertson, 1934) values, mp 74 °C, $[\alpha]_D^{32} = +121°$ (water)). (II) 2,3-Di-*O*-methyl-D-mannose, mp 107–108 °C, $[\alpha]_D^{25} = -16°$ (C 1.5, water) (cf. literature (Robertson, 1934) values, mp 106 °C, $[\alpha]_D^{25} = -15.8°$ (water)). The anilide (Hirst & Jones, 1949) had mp 136 °C. (III) 2,3,6-Tri-*O*-methyl-D-mannose, $[\alpha]_D^{25} = -11°$ (water) (cf. literature (Hirst, Hough, & Jones, 1949) values $[\alpha]_D^{25} = -10°$ (water)). The hydrazide (Hirst et al., 1949) had mp 121–131 °C. GLC of the partially methylated alditol acetates (Kapoor et al., 1998), obtained by reduction with $NaBH_4$ and acetylation of the hydrolysate of methylated seed gum, showed that 2,3,4,6-tetra-*O*-methyl-D-mannose, 2,3-di-*O*-methyl-D-mannose and 2,3,6-tri-*O*-methyl-D-mannose are present in 1.00:1.01:1.02 molar ratio. The seed gum was partially hydrolyzed with 50 mM $H_2SO_4$ for 12 h at 100 °C and the hydrolysate was subjected to paper chromatography (solvent-D). Elution of different fractions with distilled water gave D-galactose and D-mannose along with the following oligosaccharides: (I) [α-D-Galp (1 → 6)-D-Manp], mp 199 °C, $[\alpha]_D^{32} = +120.5°$ (C 1, water) cf. literature (Bailey, 1965) values, mp 200 °C, $[\alpha]_D^{32} = +121°$ (water). (II) Mannobiose [β-Manp (1 → 4)-D-Manp], mp 203–205 °C (from ethanol), $[\alpha]_D^{25} = -9°$ (water) (cf. literature (Aspinall, Rashbrook, & Kessler, 1958) values, mp 202–203 °C, $[\alpha]_D^{25} = -5.2$–$8.2°$); derivative phenyl osazone had mp 204 °C (cf. literature (Srivastava & Singh, 1967) values 203–206 °C). (III) Galactosyl mannobiose [α-D-Galp (1 → 6)-β-D-Manp (1 → 4)-D-Manp], mp 227 °C, $[\alpha]_D^{25} = +92$–$93°$ (C 1, water) (cf. literature (Bailey, 1965) values 228–229 °C), $[\alpha]_D^{25} = +93.3$–$94.4°$ (water).

Viscosity measurements were carried out with, No. 100, Ostwald's Viscometer tubes after 24 h of hydration of the gum. To prepare gum solutions, a weighed quantity of the gum was dissolved in minimum quantity of water then it was made up to a desired concentration and agitated vigorously for about 15 min till the solution become viscous and homogeneous. The Viscometer was thermostated to a temperature of 35 ± 0.5 °C. The pH of the gum solution was changed by adding 6N HCl and 1% NaOH.

For complexing 0.3 g of AR Borax per 100 ml of the gum solution was used after vigorous stirring for 5 min. To study gel formation with transition metal ions, 1% solution of $OsO_4$ and Fehling solutions were used. The gum does not gel on addition of $FeCl_3$ solution.

### 2.4. Graft co-polymerization of the gum

A calculated amount of purified gum, acrylamide, ascorbic acid, and silver nitrate was taken in 25 ml water in a 250 ml conical flask and thermostated at temperature (35 ± 0.2 °C). After 30 min a definite quantity of potassium persulphate was added. This time was taken as zero time and the reaction was allowed to proceed for 50 min. The separation of gum-*g*-polyacrylamide, from polyacrylamide, formed in the system was done (Bajpai & Sandeep, 1988) and the grafted gum was

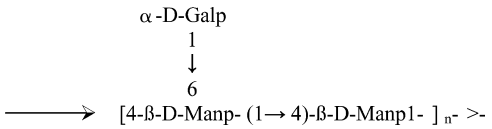


Fig. 1. Structure of seedgum from *Ipomoea turpethum*.

used for viscosity measurements. The grafted polymer was characterized by IR and thermogravimetric analysis.

## 3. Results and discussion

A water-soluble seed gum was extracted from defatted seeds with 1% aqueous acetic acid by repeated precipitation with 95% ethanol in 23% yield. It was purified by barium complexing, dialysis, and filtration through various millipore membranes. The pure polysaccharide had $[\alpha]_D^{25} = +58°$ (water), ash content 0.28% and negligible percentage of acetyl, methoxyl and uronic acid. Complete acid hydrolysis yielded D-galactose and D-mannose. The ratio of the constituent monosaccharides was found to be 1.01:2.03 by the GLC. Graded hydrolysis resulted into the preferential release of D-galactose indicating its peripheral position as end groups. Fully methylated seed gum $[\alpha]_D^{25} = +40°$ (Chloroform) on hydrolysis yielded 2,3,4,6-tetra-*O*-methyl-D-galactose, 2,3,6-tri-*O*-methyl-D-mannose and 2,3-di-*O*-methyl-D-mannose. GLC of the alditol acetates of the methylated monosaccharides showed them to be present in equimolar ratio. On oxidation with sodium metaperiodate seed gum consumed 0.8200 moles of the oxidant and liberated 0.1950 mol of formic acid per 100 g of the seed gum, which corresponded to 31.62% of the end groups. Acid catalyzed partial hydrolysis of the seed gum gave mannobiose, epimelibiose, and galactosylmannobiose along with the component monosaccharides.

The above results suggest following structural pattern for the seed gum (Fig. 1).

Relative viscosity of 1% gum solution was found to be 4.28 cP at 25 °C, which is close to viscosity of the Guar gum (4.2 cP at 27 °C as measured by Ostwald's viscometer (Whistler, 1959)) after 24 h of hydration. Viscosity of fully hydrated gum solutions was found to vary directly with changes in temperature over a range of 25–40 °C. As the temperature increases, the viscosity was found to decrease. At 30 °C, the viscosity of 1% gum solution was measured to be 3.24 cP. The gum solutions were found to be stable over a wide range of pH (1–10) due to its non-ionic nature. After grafting the viscosity of the 1% gum solution at 25 °C was measured as 26.5 cP. The grafted gum was found to be more thermally stable. Grafting percentage was found to be 98%.

With borate ions, gum solutions (2% and above), cohesive gels formed, but the gel formation was pH dependent. The optimum pH was found to be 7–10. Gels formed were reversible and was liquefied by dropping the pH below 7 and by heating. Gels were also liquefied by addition of polyols, e.g. mannitol and glycerol.

With Fehling solution and 1% $OsO_4$ solutions the gum (above 2.5%) formed gels. These gels were irreversible and not formed below pH 7.

Thus, *I. turpethum* seed gum possess non-ionic characteristics of commercial seed gums like guar, carob, and locust bean gums and have potentiality to be used in industries as commercial gum. Grafting of the gum with vinyl monomers increase the viscosity tremendously and make them resistant to biodegradation. Viscosity of the grafted gum solution remains stable over longer period in comparison to the seed gum in natural form.

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