



---

## Some Physicochemical Properties of Crosslinked Acacia Gum

**Mbah CC\*, Ogah JA, Builders PF, Kunle OO**

Department of Pharmaceutical Technology and Raw Materials Development, National Institute for Pharmaceutical Research and Development (NIPRD), Idu, P. M. B 21 Garki, Abuja, Nigeria.

---

### ABSTRACT

Acacia gum was crosslinked using calcium chloride and some of the physicochemical properties evaluated with the aim of obtaining a polymer of improved quality for wider pharmaceutical, food and cosmetic applications. The crosslinking was done by ionic gelation with calcium chloride in distilled water and some physicochemical properties of both the natural and crosslinked acacia gum were determined. The pH, flowability and moisture sorption of the crosslinked gum were significantly ( $P < 0.005$ ) increased over those of the natural acacia gum. The crosslinked acacia gum may be more suitable for solid dosage formulations of moisture-stable drugs.

**KEYWORDS:** acacia gum, cross-linking, physicochemical properties

---

### INTRODUCTION

Acacia gum has been used in pharmaceutical formulations for many years. It is the dried exudates obtained naturally from the stems and branches of *Acacia Senegal* (L) Willdenow or the related species of acacia (Fam.; Leguminosae). The plant is majorly found in Sudan and also some areas in Chad, Senegal and Nigeria. The usage in pharmaceutical formulations is based on its natural properties of emulsification, stabilization, demulcent action, adhesiveness and binding action [1, 2]. Acacia gum or Gum Arabic, as is otherwise called, is commonly employed as binder in tablet formulations. It is also employed to suspend insoluble drugs and prevent precipitation in suspensions and emulsions. Its demulcent and soothing characteristics are utilized in many cough drops and syrups. In the cosmetic industry, it is employed as a protective colloid in the preparation of creams and lotions [1, 2].

Amongst the natural hydrocolloids, acacia possesses unique properties probably due to its extreme water solubility. Most common gums cannot be dissolved in water at concentrations higher than about 5 % due to their very high viscosities [2, 3]. Acacia gum is extremely soluble and does not form very viscous solutions/dispersions at low concentrations of 1-5

% unlike most gums. High viscosities are obtained with acacia gum at concentrations of about 40-50 % in water. Acacia gum is insoluble in oils and in most organic solvents. It is soluble in aqueous ethanol up to a limit of about 60 % ethanol while limited solubility can be obtained with glycerol and ethylene glycol [1, 3].

Cross linking of polymers is done to influence the molecular structure which affects its physical characteristics such as particle properties, hydration and water sorption capacities, to mention a few. As a method of gum modification, cross-linking involves re-enforcing bonds with chemical bridges between the molecules or polymer chains. As a result, the cross-linked gum may become more resistant to high temperature, high shear and have improved viscosity and textural properties than the natural gum. Generally, cross-linking can be achieved by physical or chemical methods. In the ionic gelation technique, the crosslinking agent induces an interaction between the ionic or functional moiety and the polymer chain leading to formation of a more strongly bonded molecular chain network [4]. The essence is to promote a positive difference in the polymers' physical properties.



\*Author for correspondence; E-mail address: [mbacc@yahoo.com](mailto:mbacc@yahoo.com) Phone: 08087716982.

Cross-linked polysaccharides may be used for coating and as matrices for embedding medicinally active substances or drugs for the purposes of controlled drug delivery. The precise chemical and molecular structure of acacia gum differs according to the botanical origin of the gum. The differences are reflected in some of the analytical properties of the gum and as a result, the functional properties and uses of acacia gum are also dependent on its source. This study is therefore designed to cross-link natural acacia gum, using calcium chloride and evaluate some of the physicochemical properties, with the aim of improving its quality for wider pharmaceutical, food and cosmetic applications.

## MATERIALS AND METHODS

### MATERIALS

Acacia gum powder (BDH, England), calcium chloride, acetone (Riedel-de Haën, Italy), and distilled water. All other reagents used were of analytical grade.

### METHODS

#### Crosslinking by ionic gelation with calcium chloride

The method of ionic gelation earlier reported was adopted [4]. A 100.0 g quantity of acacia gum powder (BDH, England) was weighed and dispersed properly in 2000.0 ml of distilled water in a conical flask by shaking for 24 h using a mechanical shaker ((Type 3017, GFL GmbH, D3006, Germany) ). One litre of 1.0 M calcium chloride solution was then added and shaken for 6 h and then allowed to stand for another 6 h. The hydrated acacia particles were then harvested by filtration using a clean muslin cloth and washed serially with cold acetone to remove any residual water. The material was then air dried, pulverized, packed in screw-capped containers and stored in a desiccator for further use.

#### Physicochemical characterization

##### pH determination

Dispersions (1 %w/v) dispersion of the natural and treated/cross-linked acacia gum were prepared, respectively and their pH measured using a pH meter (Corning, England) after first standardizing with standard buffer solutions (pH 4.0, 7.0 and 9.2, respectively). The procedure was performed thrice and the mean calculated.

##### Bulk density

The natural and the crosslinked acacia gum powder were respectively weighed and poured into a 250 ml

measuring cylinder. The volume occupied was noted and the bulk density calculated from the formula [5]:

$$\text{Bulk density (g/cm}^3\text{)} = \text{weight/volume} \dots\dots\dots (1).$$

The procedure was carried out thrice and the mean calculated.

##### Tapped density

The natural and the crosslinked acacia gum powder were respectively weighed and poured into a 250 ml measuring cylinder. The cylinder was held in place and tapped using a Stampfvolumeter (STAV 2003 JEF, Germany) until constant volume was attained (about 200 taps). The volume attained was noted and the tapped density calculated from the expression:

$$\text{Tapped density (g/cm}^3\text{)} = \text{weight/final volume} \dots\dots (2).$$

The procedure was carried out thrice and the mean calculated.

##### True density ( $D_t$ ).

The fluid displacement method was used for determination of the true density of the acacia gum powders. A density bottle was washed and dried thoroughly in an oven and weighed. The density bottle was then filled with xylene as the displacement fluid and weighed. A 1.0 g quantity of the material was weighed and poured into the xylene – containing bottle with sample and then weighed. The process was repeated twice and mean  $D_t$  calculated from the relationship:

$$D_t = w/[(a + w) - b] \times SG \dots\dots\dots (3)$$

where w = weight of the acacia gum powder = 1.0 g; a = weight of density bottle + xylene; b = weight of density bottle + xylene + acacia gum powder; SG = specific gravity of the displacement fluid, xylene = 0.863.

##### Flow Properties

The static angle of repose was measured according to the fixed funnel and free standing cone method [6]. A 50 g quantity of the dried powdered gum was carefully poured to flow through a glass funnel of orifice diameter 2 cm, clamped 10 cm above a flat surface, forming a cone. The mean diameters of the powder cone formed were measured and the internal angle of repose calculated using the equation [7]:

$$\text{Angle of repose} = \text{Tan } A = 2h/D \dots\dots (4)$$

where h = height of the powder heap (cone), D = diameter of base of the powder cone.

The Hausner ratio and compressibility indices of the dried powdered gums were determined with the

data obtained for the bulk and tapped densities using equations [1, 5, 8]:

$$\text{Compressibility (\%)} = \frac{\text{Tapped density} - \text{Bulk density}}{\text{Tapped density}} \times 100 \dots \dots (5)$$

$$\text{Hausner Ratio} = \frac{\text{Tapped density}}{\text{Bulk density}} \dots \dots (6)$$

### Moisture content

Moisture content of the natural and the cross-linked acacia gum powder was determined by slight modification of the requirement in the British Pharmacopoeia (BP) for loss on drying of extracts [9]. A clean dry Petri dish was weighed and 1.0 g of the acacia powder weighed onto the Petri dish a moisture balance [6]. The loss in weight was monitored taking note of the start and stop time and the per cent loss in weight was calculated from the expression:

$$\% \text{ weight loss} = \frac{(W_i - W_f)}{W_i} \times 100 \dots \dots (7)$$

where  $W_i$  = initial weight of the sample,  $W_f$  = final weight of the sample.

The procedure was performed thrice and the mean weight loss calculated and recorded as the moisture content.

### Foaming capacity (%)

A 1.0 g quantity of gum sample was put in 20 ml distilled water and shaken for 30 min. This was allowed to stand for 30 min and the volume of the foam formed by the gum measured. The foaming capacity was calculated using eqn. 8:

$$\text{FC (\%)} = \frac{\text{vol. of mix. after homogenization} - \text{vol. before homogenization}}{\text{Vol. before homogenization}} \times 100 \% \dots \dots (8)$$

### Emulsion capacity (%)

A 1.0 g quantity of the gum was properly dispersed in 20 ml olive oil in a test tube. This was shaken and allowed to stand for 30 min. The emulsion capacity was taken as the difference between the initial volume of oil used and the volume of clear oil standing above the sedimented gum divided by the weight of gum expressed as a percentage. The emulsion capacity was calculated using eqn. 9:

$$\text{Emulsion Capacity} = \frac{\text{vol. of emulsified oil (ml)}}{\text{weight of sample (g)}} = \frac{20 \text{ ml} - \text{vol. of oil on top}}{\text{weight of sample}} \dots \dots (9)$$

### Hydration Capacity

The method of Kornblum and Stoopak was used [10]. A 1.0 g each of the samples was placed in

each of four 15 ml plastic centrifuge test tubes and 10 ml of distilled water was added from a 10 ml measuring cylinder and then stoppered. The contents were mixed on a vortex mixer (C. 560E Vortex-Gennie Scientific Industry, USA) for 2 min. The mixture was allowed to stand for 10 min and immediately centrifuged at 2000 rpm for 5 min on a GS-15 centrifuge (Beckman, Germany). The supernatant was carefully decanted and the sediment weighed. The hydration capacity was taken as the ratio of the weight of the sediment to the dry sample weight.

### Moisture Sorption Profile

Samples (1.0 g) of the samples were accurately weighed and evenly distributed over the surface of a 70 mm tarred (aluminum foil-lined) Petri dish. The samples were then placed in a large dessicator containing distilled water (RH = 100 %) in its reservoir at room temperature. At 24 h intervals over a five day period, the weight gained by the exposed samples was recorded and the amount of water absorbed calculated from the weight difference. The percentage moisture sorption was calculated using the expression [11]:

$$\text{Moisture uptake} = \frac{M_e}{M_d} \times 100 \% \dots \dots \text{eqn. 10}$$

where  $M_e$  is the amount of moisture absorbed at equilibrium and  $M_d$  is the dry weight of the material.

## RESULTS AND DISCUSSION

Table 1 shows the results of the physicochemical properties carried out on the natural and crosslinked acacia gum. Acacia gum (Gum Arabic) is known to be naturally neutral to slightly acidic [6]. The crosslinking appears to have modified the acidity to very slightly neutral. This increase in pH may be attributed to replacement of hydrogen ion-containing moiety in the polymer chain network and may reduce the adhesive property. The use of acacia gum (or gum Arabic) as a suspension, adhesive or fixing agent or a food stabiliser or emulsifier (E414) arises from its ability to strongly adhere to liquid or solid hydrophobic surfaces [12].

The reduced bulk and tapped densities of the crosslinked polymer resulted in improved Hausner ratio and compressibility index which are considered as indirect measurements of powder flowability [9]. This may make it more useful in solid dosage formulations. Hausner ratio is indicative of interparticle friction while the Carr's index shows the ability of a material to diminish in volume [9]. As the value of these indices decrease the flow of the powder increases. In general however, Hausner ratio greater than 1.25 indicates poor flow and

Carr's compressibility index below 16 % indicates good flowability while values above 35 % indicate cohesiveness.

The hydration capacity indicates the degree to which a material is capable of absorbing its own equivalent weight of water [13]. As the gum absorbs water into its particulate/molecular network the particles swell. The bulk of the absorbed water would exist in a 'free' state in between the particles. The swollen state of hydrogels is a consequence of the balance between cohesive and hydration forces on the chains. The hydrophilic polymers swell when water molecules interact with the polar groups mainly -OH and -COOH, on the polymer backbone or side chains [8]. The crosslinked acacia gum showed less hydration capacity due probably to less interparticulate spaces arising from bridging of the ionic or functional moiety and the polymer chain by the crosslink.

If employed as a matrix, the natural gum may therefore absorb more water and cause faster delivery by capillary or wicking action of the water and swelling of the gum particles.

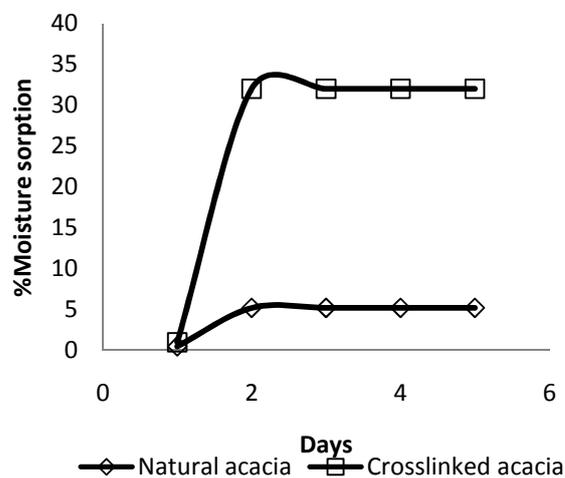
The moisture sorption profiles of the natural and crosslinked acacia gums are shown in Fig. 1. The crosslinked gum clearly showed significantly ( $P < 0.05$ ) higher water sorption than the natural gum. Moisture sorption of a material gives an indication of its moisture sensitivity which reflects the relative stability under humid conditions. From the third day, moisture sorption remained stable/constant for both natural and crosslinked acacia indicating saturation. The higher the rate of moisture sorption the higher will be the deterioration effect on the constituents of a dosage form that undergo hydrolytic decomposition [13]. Due to the high moisture sorption of the crosslinked acacia gum it may be used in tablet formulations of moisture-stable drugs.

**Table 1:** Results of some physicochemical properties of the natural and crosslinked acacia gum.

Property	Polymer Natural acacia powder	Crosslinked acacia
pH	4.730 ± 0.78	7.730 ± 0.50
Bulk density (g/ml)	0.806 ± 0.50	0.741 ± 0.53
Tapped density (g/ml)	0.955 ± 0.48	0.800 ± 0.51
True density (g/ml)	1.330 ± 0.60	1.420 ± 0.54
Hausner Ratio	1.380 ± 0.35	1.130 ± 0.50
Compressibility Index (%)	15.600 ± 0.30	7.380 ± 0.40
Hydration capacity	5.700 ± 1.20	4.900 ± 0.89
Moisture content (%)	14.000 ± 1.32	5.000 ± 0.90

Moisture sorption profile of natural and crosslinked acacia gum

## CONCLUSION



Some physicochemical properties of the natural acacia gum powder were influenced by the process of ionic gelation with calcium chloride. The pH, flowability and moisture sorption profile of the crosslinked gum were significantly ( $P < 0.005$ ) increased over the natural acacia gum. Hence, the crosslinked acacia gum will be more suitable for tablet and emulsion formulations of moisture-stable drugs.

## ACKNOWLEDGEMENT

The authors are grateful to all the technical laboratory staff of the Department of Pharmaceutical Technology and Raw Materials Development, National Institute for Pharmaceutical Research and Development (NIPRD) for their assistance and to NIPRD for provision of the facilities for this study.

## REFERENCES

- Ohwoavworhwa FO, Adeola JI. (2005). Extraction and characterization of starch obtained from earth pea. *Afr. J. Pharm Res. Dev.* 3-5.
- Iwuagwu, MA, Eba YI, Omokhafa AA, Anozie JD. (1996). Locally sourced celluloses as compressed tablet excipients. *J. West Afri. Pharm.* 10 (1): 18-24.
- Train D. (1958). Some aspects of the property of angle of repose of powders. *J. Pharm. Pharmacol.* 10, 127T-134T.
- Patil, J.S., Kamalapur, M.V.S., Marapur, C., Kadam, D.V. (2010). Ionotropic gelation and polyelectrolyte complexation: the novel techniques to design hydrogel particulate sustained, modulated drug delivery system: A review digests. *J. Nanomat. Biostructures.* 5(1), 241-248.
- Well, J. In: *The Science of Dosage Form Design*, Aulton, M. E. (Ed.), Churchill Livingstone, Toronto, 2003, 2<sup>nd</sup> ed. pp. 113-138.

6. The British Pharmacopoeia. 2004. Vol. IV. A252, A256-272. Her Majesty's Stationary Office, London.
7. Carter SJ. Powder Flow and Compaction: In Cooper and Gun's Tutorial Pharmacy. 2<sup>nd</sup> Ed. CBS Publishers and Distributors, India 1986; 186-232.
8. BuildersPF, Mbah CC, Attama AA. (2011). Critical intrinsic and functional properties of a novel gelling polysaccharide from *Diocleareflexa* with drug delivery potential. BJPR (in press).
9. Staniforth JN. (1996). Powder flow. In: Aulton ME. (Ed.) Pharmaceutics: The science of dosage form design. Churchill Livingstone. Edinburgh, London. 600-615.
10. Kornblum SS, Stoopak SB. (1973). A new tablet disintegrant agent: cross-linked polyvinylpyrrolidone. J. Pharm. Sci. 62 (1): 43-49.
11. Lin YC, Chen X. (2005). Moisture sorption-desorption characteristics and its effect on the mechanical behavior of the epoxy system. Polymer, 46 (5):11994-12003.
12. Renard D, Lavenant L, Ralet M-C, Sanchez C. (2006) Acacia senegal gum: a continuum of molecular species differing by their protein to sugar ratio, molecular weight and charges Biomacromolecules 7, 2637-2649.
13. Ohwoavworhua FO, Kunle OO, Ofoefule SI. (2004). Extraction and characterization of microcrystalline cellulose derived from *Luffacylindrica* plant. Afr. J. Pharm Res. Dev. 1-5.

