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Extraction and Characterization of *Artocarpus integer* Gum as Pharmaceutical Excipient

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A – research concept and design; **B** – collection and/or assembly of data; **C** – data analysis and interpretation;
D – writing the article; **E** – critical revision of the article; **F** – final approval of the article

Abstract

Background. Natural polymers are widely used as excipients in pharmaceutical formulations. They are easily available, cheap and less toxic as compared to synthetic polymers.

Objectives. This study involves the extraction and characterization of kathal (*Artocarpus integer)* gum as a pharmaceutical excipient**.**

Material and Methods. Water was used as a solvent for extraction of the natural polymer. Yield was calculated with an aim to evaluate the efficacy of the process. The product was screened for the presence of Micrometric properties, and swelling index, flow behavior, surface tension, and viscosity of natural polymers were calculated.

Results. Using a water based extraction method, the yield of gum was found to be 2.85%. Various parameters such as flow behavior, organoleptic properties, surface tension, viscosity, loss on drying, ash value and swelling index together with microscopic studies of particles were done to characterize the extracted gum. The result showed that extracted kathal gum exhibited excellent flow properties. The gum was investigated for purity by carrying out chemical tests for different phytochemical constituents and only carbohydrates were found to be present. It had a good swelling index (13 ± 1). The pH and surface tension of the 1% gum solution were found to be 6 ± 0.5 and 0.0627 J/m^2, respectively. The ash values such as total ash, acid insoluble ash, and water soluble ash were found to be 18.9%, 0.67% and 4% respectively. Loss on drying was 6.61%. The extracted gum was soluble in warm water and insoluble in organic solvents. The scanning electron micrograph (SEM) revealed rough and irregular particles of the isolated polymer.

Conclusions. The results of the evaluated properties showed that kathal-derived gum has acceptable pH and organoleptic properties and can be used as a pharmaceutical excipient to formulate solid oral dosage forms (**Polim Med. 2014, 44, 2, 69–74**).

Key words: kathal, gum, pharmaceutical excipient, natural polymer.

Gum and mucilage are natural polymers widely used as conventional and novel dosage forms. Natural polymers are generally safe for pharmaceutical formulation. They are high molecular weight; water soluble polymers are made up of a monosaccharide unit and joined by a glucosidic bond [1]. Gummy excaudate of natural polymers such as protein, enzyme, muscle, fiber, polysaccharides have been used to formulate various pharmaceutical products [1, 2]. The well known natural polymers are gelatine, aloe mucilage, guar gum, gum karaya, bhara gum, sodium alginate, locust bean gum, okra mucilage, gum acacia, linseed mucilage, etc. These natural polymers are applicable in different pharmaceutical dosage forms like matrix controlled systems, microspheres, nanoparticles, buccal films and semi solid formulations [3, 4]. Hence, by modification, gums can alter their physicochemical properties. Mucilage is a metabolized product which is intracellularly formed without injury to the plant [5]. Gums are readily soluble in water while mucilage forms a slimy mass in the presence of water. Gum and mucilage are translucent, amorphous substances which are produced by plants as a protection during injury. Gum, cellulose, mucilage and pectin are distinguished by the condensation of hexose and pentose sugar [6, 7]. Gum and mucilage can be obtain from the middle lamella as in algae, the cell wall as in the seed epidermis as well as endodermis, some secretory cells as in squill and are also present in the schizogenous sacs [8]. Gum has various pharmaceutical applications such as a suspending agent for insoluble solid components in a mixture, an emulsifying agent for the oil phase and an adhesive in troche masses and pills [8, 9]. Natural polymers have found applications in households, agriculture the food industry, and in packaging it helps in decreasing environmental pollution and disposal in landfills [10].

Materials and Methods

Extraction procedure: Kathal (*Artocarpus integer*) was collected from a local market in Greater Noida, India. The collected kathal was carefully washed and dried in a shaded place for 24 h and further dried at 30–40 °C until a constant weight was obtained. The size was reduced through a grinder. The powdered fruit passed through a no. 22 sieve and was stored in an airtight container for further use. Extraction of the gum included two steps.

Step 1: extraction of gum: As the authors described elsewhere, the powdered fruit was put in 500 mL of distilled water in a 1000 mL beaker, then heated and stirred continuously at 60 °C for approximately 4 h. The concentrated solution was filtered through muslin cloth and cooled at 4–6 °C [11].

Step 2: Isolation of Gum: the extract was poured into ethyl alcohol, thus precipitation of the gum occurred. This allows the filtration through muslin cloth. Washed with ethyl alcohol and precipitated, the gum has collected through the muslin cloth by filtration. The gum was further dried to a constant weight at 35–45 °C in a hot air oven. The hard gum cake was ground and sieved through a no. 22 sieve and stored in a desiccator for further use [12].

Determination of carbohydrate presence in kathal gum: an aqueous extract was mixed with Molisch's reagent and followed by the addition of sulphuric acid. The violet color ring appeared at the junction, showing the presence of carbohydrates [13, 14].

Determination of purity of kathal gum: to measure the purity of the extracted gum, tests for alkaloids, proteins, gum, fats, tannins and amino acids were performed [13, 14].

Organoleptic evaluation of isolated gum: isolated gum was characterized for organoleptic properties such as color, odor, taste, fracture and texture [15].

Ash values: ash values such as total ash, acid insoluble ash and water-soluble ash were determined using equations 1, 2 and 3, respectively [16].

$$\textit{total ash value} = \frac{\textit{weight of ash}}{\textit{weight of polymer}} \times 100 \quad (1)$$

$$\textit{acid insoluble ash} = \frac{\textit{weight of acid insoluble ash}}{\textit{weight of drived powder}} \times 100 \quad (2)$$

$$\textit{water soluble ash} = \frac{\textit{weight of water soluble ash}}{\textit{weight of drived powder}} \times 100 \quad (3)$$

Solubility behavior: The dry gum powder was shaken with different solvents and further solubility was determined [16].

pH of gum: the gum was weighed and dissolved in water separately to get a 1% w/v solution. The pH of the solution was determined using a digital pH meter [17].

Swelling index: the swelling index was calculated as per equation 4 [17].

$$\textit{swelling indes} = \frac{\textit{final volume} - \textit{initial volume}}{\textit{final volume}} \times 100 \quad (4)$$

Surface tension: the surface tension of the selected gum was determined by the drop weight method, using a stalagmometer. The surface tension of the polymer has been reported to influence the binding quality of the polymer. Surface tension was calculated as per equation 5 [17, 18].

$$\sigma_{solution} = \sigma_{water} \frac{m\ (solution)}{m\ (water)} \quad (5)$$

where:
$\sigma_{surface}$ = surface tension of the solution
$\sigma_{surface}$ = surface tension of water
m (solution) =weight of solution
m (water) = weight of water

Viscosity: The viscosity of the kathal gum was determined using an Oswald viscometer. The calculations were done using equation 6 [19].

$$\eta_s = \eta_w \times \frac{t_s\, \rho_s}{t_w\, \rho_s} \quad (6)$$

where:
η_s = viscosity of solution
η_w = viscosity of water
t_s and t_w = time of solution and water respectively
ρ_s and ρ_w = density of solution and water

Loss on drying: One gram of powder was weighed accurately in a weighing bottle and was dried in a hot air oven at 105 °C and the weight was checked at intervals of 10 min, until a constant weight was obtained. The percentage of weight lost by the powder was calculated using equation 7 [19].

$$\textit{loss on drying} = \frac{\textit{initial weight} - \textit{final weight}}{\textit{initial weight}} \times 100 \quad (7)$$

Bulk density and bulkiness: the inverse of bulk density is called bulkiness. An accurately weighed quantity (50 g) was introduced into a graduated measuring cylinder. The cylinder was fixed on the bulk density apparatus and the volume occupied by the powder was noted. Then, the powder was subjected to tapping in a bulk density apparatus until a constant volume was obtained. The final volume (bulk volume) was noted.

Bulk density, tapped density and bulkiness were calculated using equations 8–10, respectively [18–20].

$$bulk\ density = \frac{weight\ of\ powder\ blend}{weight\ of\ appatrent\ volume} \tag{8}$$

$$tapped\ density = \frac{weight\ of\ powder\ blend}{tapped\ volume} \tag{9}$$

$$bulkiness = \frac{1}{bulk\ density} \tag{10}$$

True density: among the various methods available for the determination of true density, the liquid displacement method is the simplest method and was used in the present study. Acetone was selected as the liquid for displacement, because gum is insoluble in acetone [20, 21].

Powder flow property: this property is also known as compressibility. The finely powdered gum (5 g) was transferred into a measuring cylinder and calculations were done using bulk density apparatus [21].

$$\mathrm{T}^{an\theta} = \frac{h}{r} \tag{11}$$

where:
θ = angle of repose
h = height of pile
r = radius of pile

Powder cCompressibility (Carr's Consolidation Index): this property is also known as compressibility. Carr's consolidation index was calculated using equations 12 and 13 [21].

$$Carr's\ index = \frac{tapped\ density - bulk\ density}{tapped\ density} \times 100 \tag{12}$$

$$Hausner's\ ratio = \frac{tapped\ density}{bulk\ density} \times 100 \tag{13}$$

Particle size analysis: particle size distribution of the gum was determined by the optical microscopy method. The diameters of 600 particles were measured using a calibrated eye piece micrometer, which was calculated as per equations 15 and 16 [21].

$$\begin{matrix} size\ of\ individual \\ particle \end{matrix} = \begin{matrix} no.\ of\ individual\ in\ eye\ piece \times \\ \times\ calibration\ factor \end{matrix} \tag{15}$$

$$calibration\ factor = \frac{stage\ reading \times 0.01}{ovular\ reading} \tag{16}$$

IR Spectrum: the IR spectrum of *Artocarpus integer* gum was taken using a Bruker ATR spectrophotometer (Model-ALPHA, Laser class 1, Serial number 200301, made in Germany).

Results and Discussion

After extraction and further precipitation by ethyl alcohol, the yield of the gum was 2.85% w/w obtained. For a determination of purity of the isolated kathal gum, different phytochemical tests were carried out including Molisch's test, Benedict's test, Barfoed's test and Seliwanoff's test. This showed that kathal gum has passed the carbohydrate test. Confirmation of gum was done when it gave a negative result for tannins, alkaloids and proteins. This can be considered proof of the purity of the isolated gum, as depicted in Table 1.

Table1. Determination of purity of isolated gum

S. No.	Test	Present/absent
1.	Carbohydrates	+
2.	Hexose Sugar	+
3.	Monosaccharides	–
4.	Proteins	–
5.	Fats and oils	–
6.	Tannins and Phenolic Compounds	–
7.	Alkaloids	–
8.	Amino acids	–
9.	Mucilage	–

+ present, – absent.

The results for loss on drying showed a value of 6.61%. This indicates that the gum is hygroscopic in nature and needs to be stored in an air-tight container. Kathal gum was found to be soluble in warm water, slightly soluble in cold water and insoluble in benzene, ether, chloroform, n-butanol, ethanol, acetone, glycerine and paraffin. It had a good swelling index (13 ± 1) and good water uptake capacity. The pH of a 1% solution of kathal gum was found to be 6 ± 0.5 and its surface tension was found to be 0.0627 J/m^2. Other phytoconstituents were absent in the isolated powder. The particle size of the polymer was found to be 45 µm. The result obtained of kathal gum and it was observed that the gum is a brownish color, odorless, tasteless, rough and irregular in shape. The ash values were calculated to characterize gum; total ash, acid insoluble ash and water soluble ash were found to be 18.9%, 0.67% and 4%, respectively. A physical characterization of the gum was carried out for bulk density and bulkiness, true density and powder flow behavior. The bulkiness value indicated that the powder is 'light' in nature. The value of Carr's index indicated that the powder has poor flow character. The results obtained in the micromeritic characterization of the gum are shown in Table 2.

A Bruker ATR (Model-ALPHA, Laser class 1, Serial number 200301, made in Germany) spectra showed that kathal gum contains carboxylic acid, amide, alk-

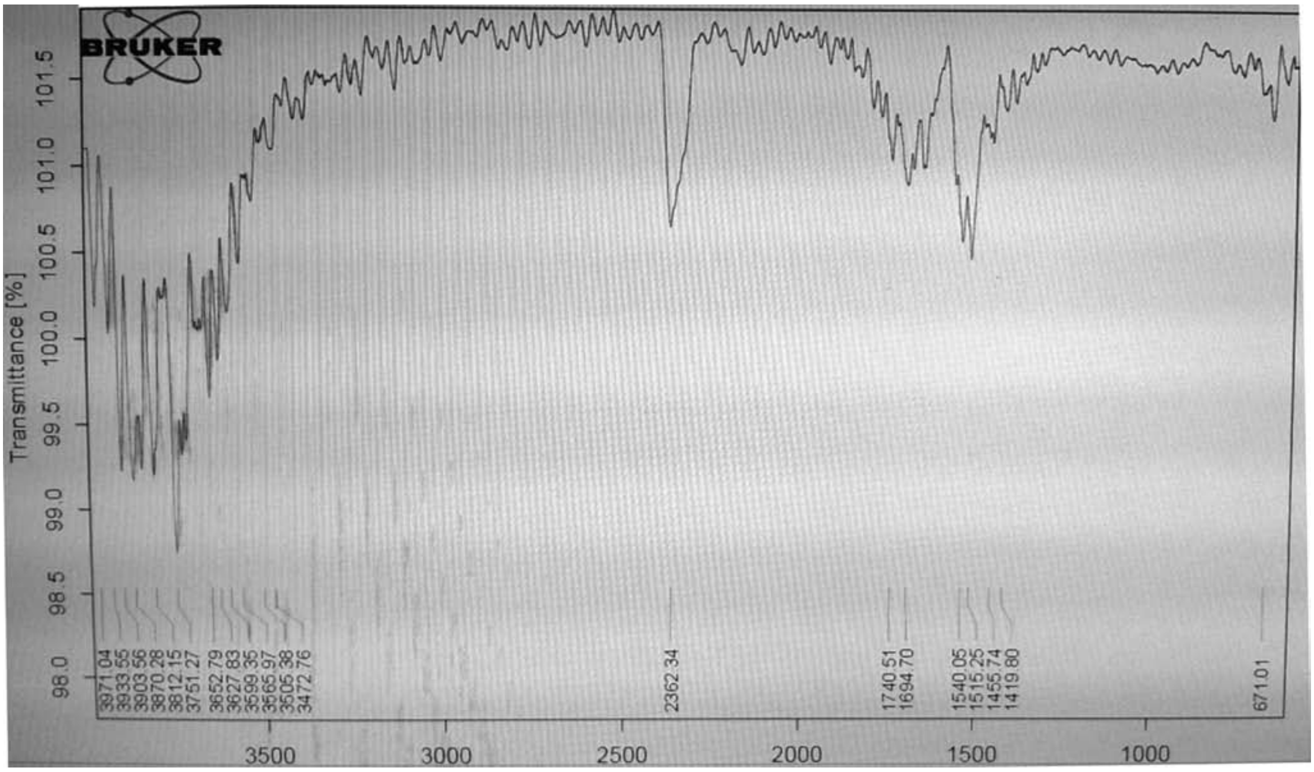

Fig. 1. IR spectra of kathal gum

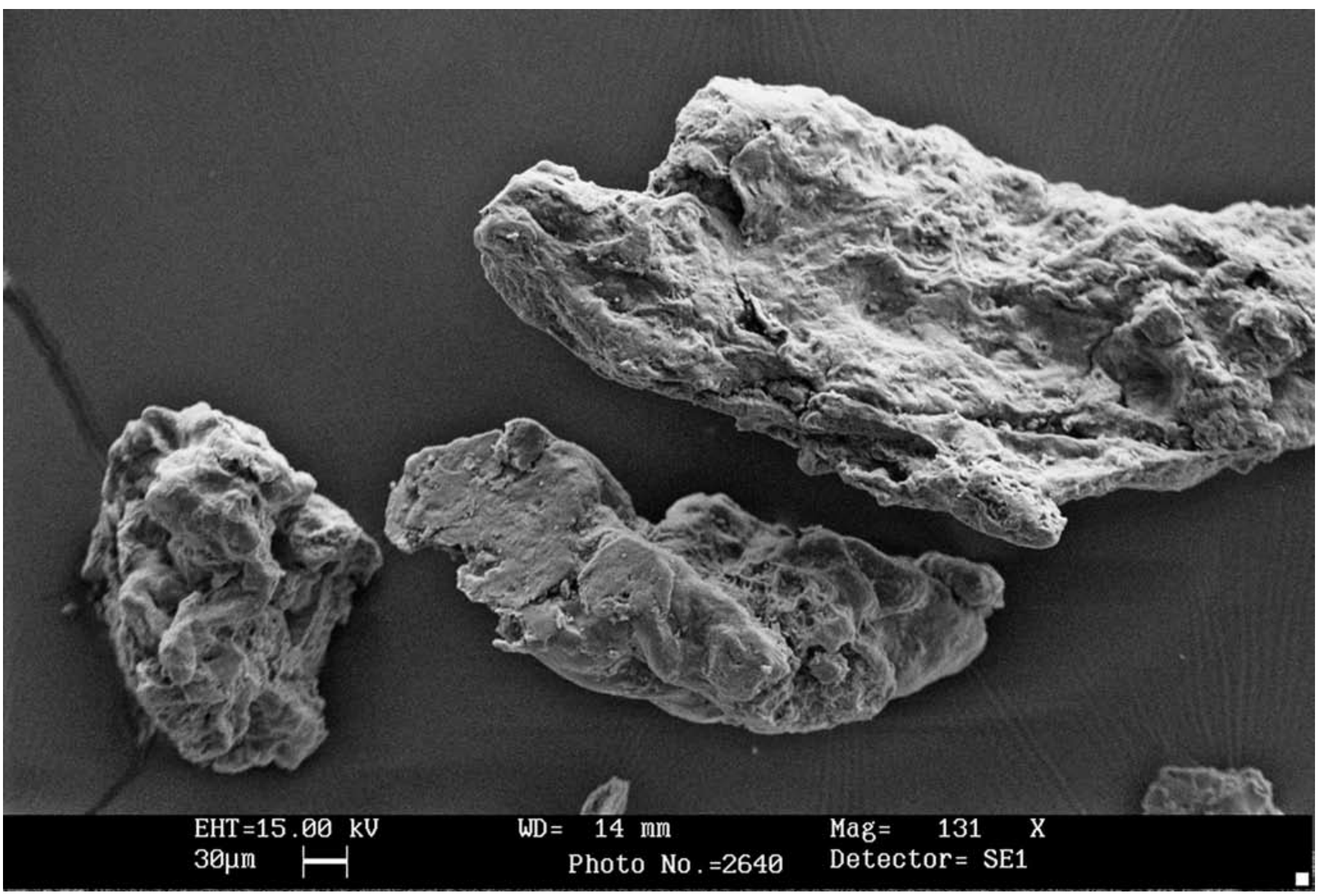

Fig. 2. Scanning electron micrograph of *Artocarpus integer* gum

Table 2. Micromeritic study data of the gum

S. No.	Parameters	Values[#]
1.	Angle of repose (°)	28.45 ± 1.29
2.	Carr's index (%)	31.35 ± 0.78
3.	Tapped density (g/cm^3)	0.547 ± 0.057
4.	Bulk density (g/cm^3)	0.399 ± 0.034
5.	Bulkiness (cm^3/g)	2.53 ± 0.288
7.	Particle size (µm)	260 ± 1
8.	Hausner's ratio	1.35 ± 0.026
9.	Swelling index	13 ± 1

[#] values with "±"show standard deviation and all measurement were done in triplicate studies.

ene and the hydroxyl group as shown in Fig. 1, and the peaks of these groups are shown in Table 3.

Table 3. IR study data of kathal gum

S. No.	Functional group	Peak
1.	O–H	3627.83
2.	Amide	1540.05
3.	Carboxylic acid	1740.51
4.	CH_2–CH_2	1694.70

The SEM photograph, as shown in Fig. 2, of the *Artocarpus integer* gum revealed that the surface of the particles was found to be rough and irregular. Earlier, it had been reported that the rough surface of the polymer can retard the drug release from the dosage form due to the entrapment of the drug in the pores.

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