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Isolation and characterization of gum from *Chrysophyllum albidum* fruits as pharmaceutical excipient

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Abstract

This study describes the morphology, physicochemical and compressional characteristics of a natural gum derived from the fruits of *Chrysophyllum albidum.* Preliminary phytochemical screening and physicochemical properties of *Chrysophyllum albidum* gum (in comparison with tragacanth gum) were determined while scanning electron microscopy (SEM), Fourier transform infra red (FTIR) and differential scanning calorimetry (DSC) were used to characterize the morphology of the gum. The compressional behaviour of the gum in comparison with that of corn starch BP was analyzed using density measurements and indices of plasticity from the Heckel and Kawakita equations. *Chrysophyllum albidum* gum contains saponins, polysaccharide, flavonoids, tannins and non- reducing sugar. The pH of 1% w/v suspension of *Chrysophyllum albidum* was 4.13. The angle of repose of *Chrysophyllum albidum* gum was high and indicative of a slightly poor flow. FTIR Spectra reveal absorption peaks between 1638 and 1454 cm-1 while the absorption bands between 800 and 1200 cm^{-1} represent the fingerprint region for carbohydrates. The SEM images reveal that the particles are round and regular in shape while the DSC thermogram shows a gradual increase in heat flow which suggests that *Chrysophyllum albidum* gum exhibits only the amorphous structure. *Chrysophyllum albidum* is a Type C material which exhibits a slower onset of plastic deformation than corn starch but had a lower overall amount of plastic deformation during the compression process. The results establish the fundamental characteristic of *Chrysophyllum albidum* gum and explain the rationale behind its use as raw material in pharmaceutical formulations

Keywords: *Chrysophyllum albidum*; Gum; Excipients; Compressional properties

INTRODUCTION

Natural gums have found wide application in pharmacy because of their availability, eco-friendliness, low cost, low toxicity, biocompatibility, chemical modification capability and potential biodegradability. Thus, these natural polysaccharides are amongst the richest renewable source for sustainable supply of cheaper pharmaceutical products [1]. They have been widely employed in drug delivery systems as binders, disintegrants, suspending, emulsifying, thickening, film coating agents and polymer matrices in sustained release formulations. They have also been used in microspheres and nano drug delivery systems. Natural gums are high molecular weight hydrophilic carbohydrate polymers which are made up of monosaccharide units linked by glucosidic bonds. They have been isolated from *Abelmuscus esculentus, Gelidium amansii, Albizia zygia, Chondrus cryspus,*

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Cassia tora Linn, *Cyamompsis tetraganolobus, Grewia mollis* and *Chrysophyllum albidum* [2-6]. With the increase in demand for natural hydrocolloids, it has become important to look for newer sources to meet industrial demand [7].

Chrysophyllum albidum belongs to the Sapotaceae family. The fruit is almost spherical, with a slight point at the tip. There are 3 to 5 brown, shiny seeds (1-1.5 × 2 cm), with a hard coat arranged in a star-shaped pattern in the yellow pulp in the fruits. The fruit turns from greenish grey when immature to orange, pink or yellow when ripe [8]. The bark is used for the treatment of yellow fever and malaria while the leaf has anti-platelet and hypoglycemic properties [9]. The root bark has been reported to have anti-fertility effects while the stem bark extracts have antimicrobial effects [11]. Bakre and Ajakore [2] have reported the use of the gum extracted from the fruits as a suspending agent and binder.

The characterization of pharmaceutical materials prior to their use as excipients is highly essential and has assisted in the design of drug formulations to obtain a desired set of performance properties. A survey of the literature shows that there is no report of a comprehensive characterization of the gum. Therefore, the objective of this study is to isolate and characterize *Chrysophyllum albidum* gum with a view to establishing its potentials as raw material and excipient in food and drug industries.

EXPERIMENTAL

Extraction of gum. Ripe *Chrysophyllum albidum* fruits were sundried for three weeks and the seeds were removed. The fruits were then size reduced. The powder was then sifted using a sieve of size 250um. 10kg of dried powder were extracted exhaustively using 96% ethanol. The extracted gum was dried in a desiccator and milled.

Physicochemical characterization. Phyto-chemical screening was carried out using official methods in the British Pharmacopoiea [12]. The determination of the particle density was carried out using the pycnometer method and the displacement liquid used was xylene. The bulk density of *Chrysophyllum albidum* and tragacanth gums at zero pressure were obtained using the method of Kumar and Kothari [13] while the tapped density was determined by tapping the powder bed 300 times at the rate of 38 taps per minutes [14] and then repeated with 700 taps. The final tapped volume was obtained when the difference between 200 times taps is less than 2% [15].The pH of 1% w/v dispersion of the gum in water was determined using a digital pH meter. The moisture content was determined by the gravimetric method while the funnel method [16] was employed in determining the angle of repose. The mean of three determinations was taken. The swelling capacity was determined using the method of Kumar and Kulkarni [17] with slight modification while the hydration capacity was determined as per the method of Komblum and Stoopak [18].

Determination of Morphology

FTIR spectroscopy. The *Chrysophyllum albidum* gum was dried and stored in a desiccator prior to FTIR analysis. FTIR spectra were obtained using potassium bromide discs prepared from a blend of gum and dry potassium bromide on a FT-IR spectrophotometer (Mattson Galaxy 3020, England).

Scanning electron microscopy. Scanning electron microscopy (SEM) of the *Chrysophyllum albidum* gum was performed using a ZEISS EVO18 (Germany). The gum was mounted in double sided adhesive conductive carbon tape over an aluminum sample holder and sputter-coated with a gold-plasma in a sputter coater Emitech SC7620 (Germany) for 90 s with 7 mA current.

Differential scanning calorimetry. Differential scanning calorimetric (DSC) measurements of the gum sample were carried out on a Perkin Elmer Instrument (DSC 6000, USA). Test samples weighing 4-10 mg were compressed into pellets in an aluminium pan. The study was performed at heating rate of 20^{o}C/min from 30 to 430^{o}C in a nitrogen atmosphere with a flow rate of 20 mLmin^{-1}. An empty aluminum pan was used as a reference and thermograms were obtained.

Determination of compressional characteristics. Compacts of *Chrysophyllum albidum* gum were produced on a tablet machine (Model C, Carver Inc U.S.A) fitted with a pressure gauge. Prior to each compression, the flat faced punches and the die (12.5 mm) were lubricated with 2% magnesium stearate dispersion in benzene. Upon ejection, the tablets were kept over silica gel for 24 hrs to give room for elastic recovery and subsequently evaluated.

Compaction Data Analysis

Heckel plot. The predominant mode of consolidation in the *Chrysophyllum albidum* gum was assessed by Heckel analysis [19] using equation (1).

$$ln\ 1/1\text{-}D = KP + A \quad \ldots\ldots\ldots\ldots (1)$$

Py which is the mean yield pressure was obtained as the inverse of the slope (K) of the linear portion of the curve. The value of the total pre-compression densification, D_A, was obtained from the intercept on the ordinate of the linear part of the plot using equation (2).

$$D_A = 1\text{-}e^{-A} \quad \ldots\ldots\ldots\ldots\ldots\ldots (2)$$

D_B which represents the relative density from the phase of particle slippage and re-arrangement was then obtained as the difference between D_A and D_o (precompression density of the sample at zero pressure).

Kawakita plot. The degree of volume reduction, C of powders under applied pressure, P during compression was assessed by the Kawakita equation [20]:

$$P/C = P/a + 1/ab \quad \ldots\ldots\ldots\ldots (3)$$

The parameters obtained from the slope (1/a) and intercept (1/ab) were used to calculate the value of b. The constant, a, is the porosity of the powder bed prior to compression while b is related to the plasticity of the material. It should be noted however that the real physical definition of these constants is not clear [21]. D_I $(1 - a)$ is the initial relative density of the material when little pressure or tapping is applied [22] while P_K, derived from b indicates the pressure required to produce 50 % volume reduction of the powder bed [23] is used to characterize the compression behaviour of the powders.

RESULTS

Preliminary phytochemical screening reveals the presence of saponins, flavonoids, tannins, polysaccharide and non- reducing sugar. The result of the physicochemical characterization is presented in Table 1. The powder porosity, tapped and bulk densities values were higher for *Chrysophyllum albidum* gum than tragacanth gum. The swelling index and hydration capacity were comparatively lower in *Chrysophyllum albidum* gum while the moisture content was much higher. The pH of 1% suspension of *Chrysophyllum albidum* was 4.13 which is slightly acidic. The FTIR spectrum of the *Chrysophyllum albidum* gum is shown in figure 1. There is a wide band at 3445 cm-^{1} and absorption peaks between 1638 and 1454 cm^{-1}. Figure 2 shows the scanning electron microscopic image of *Chrysophyllum albidum* gum while the DSC thermogram presented in Figure 3 reveals a gradual increase in heat flow which suggests that *Chrysophyllum albidum* gum exhibits only the amorphous structure. Figures 5 and 6 show the Heckel and Kawakita plots respectively. The shape of the Heckel plots for *Chrysophyllum albidum* gum is indicative of Type C materials which principally undergo deformation by plastic flow and asperitic melting while corn starch is a Type A material. Table 3 shows that

Chrysophyllum albidum gum had higher P_y and P_k values than corn starch.

Table 1: Powder characterization

Physicochemical properties	*Chrysophyllum albidum* gum	Tragacanth *gum*
Bulk Density (g/cm^3)	0.5156	0.367
Tapped Density (g/cm^3)	0.6260	0.508
Angle of repose (^{0})	32.7	26.1
Hausner ratio	1.21	1.38
Compressibility index (%)	17.64	27.76
Powder Porosity (%)	67.15	25.67
Swelling index	0.70	4.80
Hydration capacity	1.87	2.12
Moisture content (%)	6.0	0.76
Solubility	Slightly soluble in water. Practically insoluble in acetone, chloroform, dichloromethane.	Slightly soluble in water. Practically insoluble in acetone, chloroform, dichloromethane.
pH	4.13	5.40

Table 2: Thermal properties of *Chrysophyllum albidum* gum

Parameters	*Chrysophyllum albidum* gum
Peak temperature (^{0}C)	60
Onset temperature (^{0}C)	40
Offset temperature (^{0}C)	67
Delta Cp (J/g K)	0.7

Table 3: Parameters obtained from Heckel and Kawakita plots

Sample	Heckel				Kawakita		
	D_o	P_y	D_A	D_B	Pk	A	D_I
CAG	0.362	1004.0	0.740	0.378	2.65	1.017	0.024
CS	0.400	500.0	0.222	0.447	1.05	1.023	0.018

CAG: *Chrysophyllum albidum* gum CS: corn starch

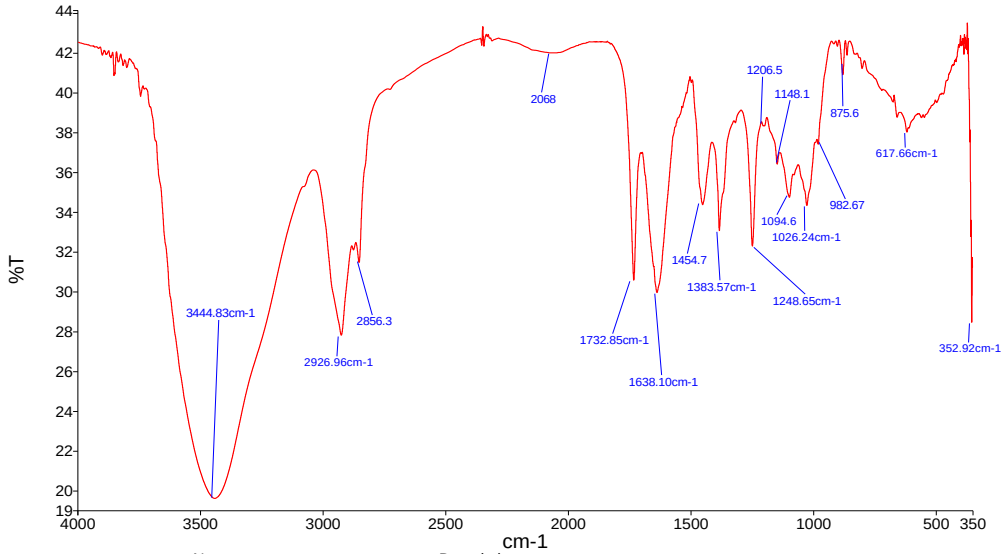

Figure 1: FTIR Spectrum of *Chrysophyllum albidum* gum.

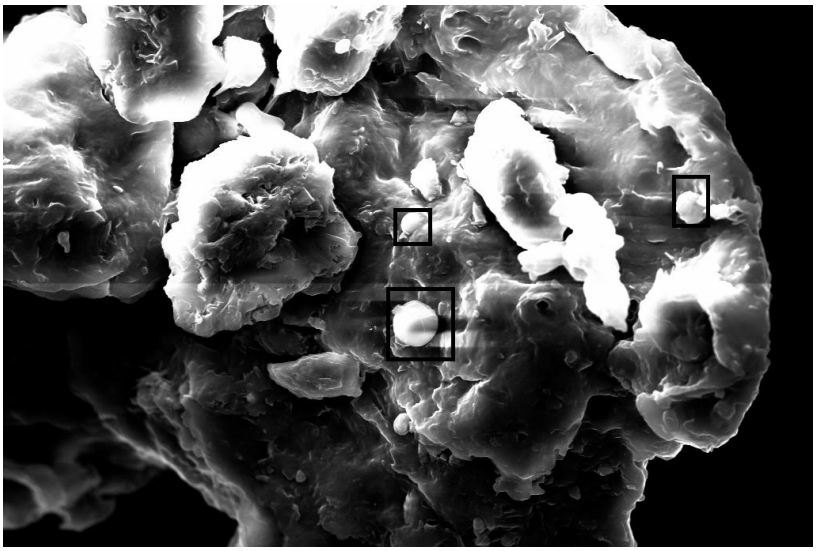

Figure 2: Scanning Electron Micrograph of *Chrysophyllum albidum* gum powder at 770X Magnification

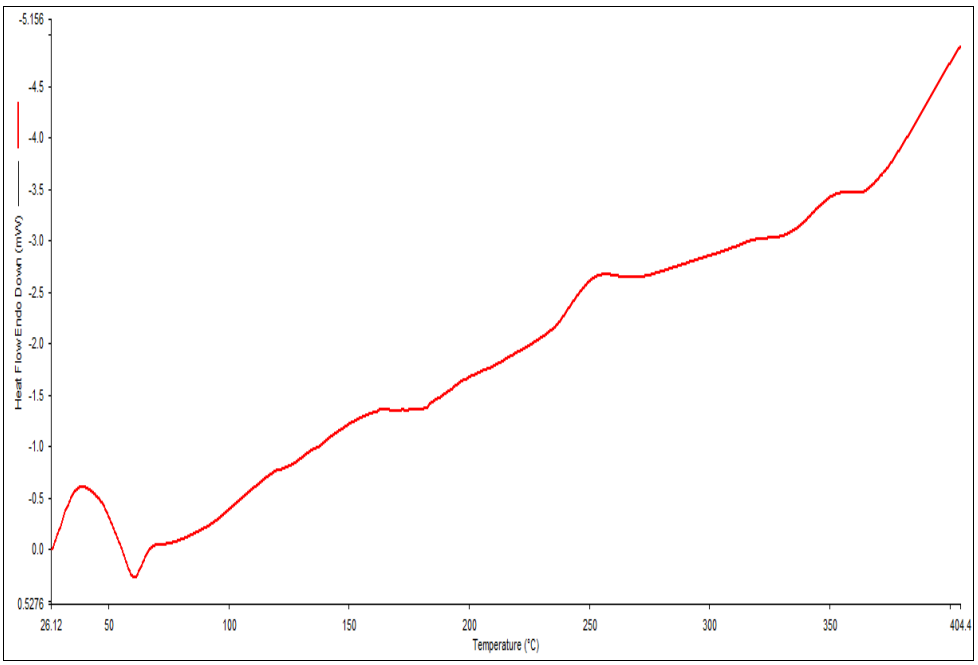

Figure 3: Differential Scanning Calorimetry thermograph of *Chrysophyllum albidum* gum

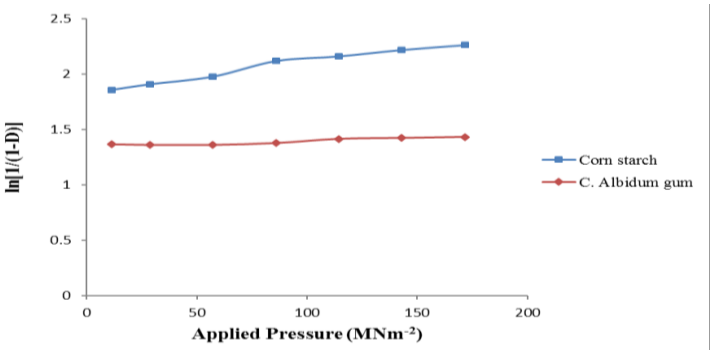

Figure 4: Heckel plots for corn starch BP and *Chrysophyllum albidum* gum

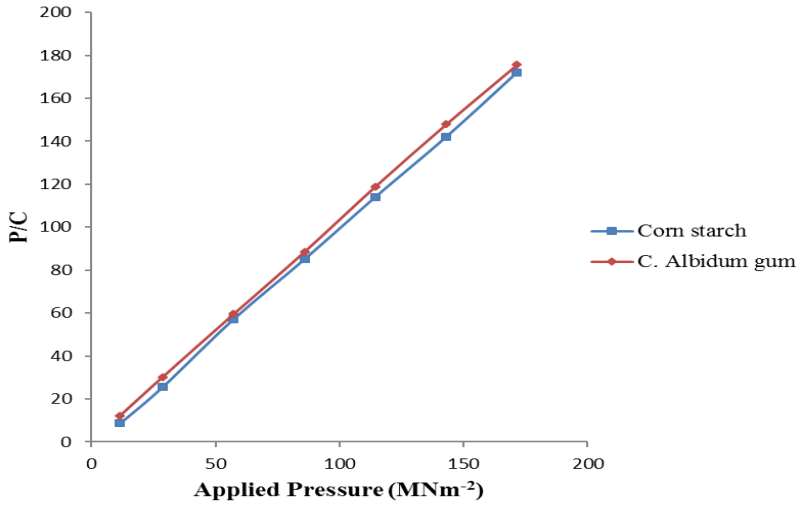

Figure 5: Kawakita plots for corn starch BP and *Chrysophyllum albidum* gum

DISCUSSION

Powder characterization. The tapped and the bulk densities are a reflection of the flow properties, arrangement and packing of the particles and therefore, the compressibility of the powder [24]. The bulk density of a powder indicates the packing characteristic during the different unit operations involved in tabletting such as mixing, filling of the die, granulation and compaction. High value of bulk density is desirable during tabletting because of a decrease in the die fill volume. The *Chrysophyllum albidum* gum is slightly soluble in water but practically insoluble in dichloromethane, absolute ethanol, acetone and chloroform. The low solubility may be because a large proportion of the gum comprises of insoluble cell wall materials. The pH of an excipient is an important factor in determining its suitability in formulation since stability and physiological activity of most preparations depend on the pH [25]. The pH of 1% suspension of *Chrysophyllum albidum* was 4.13 which is slightly acidic and thus might require adjustment if it is to be employed in the formulation of oral and buccal drug delivery systems. The swelling and the hydration capacities have significant effect on the disintegrating properties [26]. They have therefore been used to assess the swelling behavior of pharmaceutical materials which is widely regarded as a reflection of tablet disintegrating ability [27]. The result shows that *Chrysophyllum albidum gum* is able to absorb about twice its own weight of water. The swelling capacity of *Chrysophyllum albidum* gum which is an index of the increase in volume following water absorption is relatively lower than in tragacanth gum. *Chrysophyllum albidum* gum would likely produce tablet disintegration through water uptake by capillary action. The high porosity of *Chrysophyllum albidum* gum will probably assist upward intake of water during capillary process. Tragacanth gum is likely to produce better disintegrant activity than *Chrysophyllum albidum* gum due to its superior swelling and hydration capacities ($p< 0.05$) The economic importance of an excipient lies not only on the cheap and easy availability of the biomaterial but on the optimization of production processes such as during packaging and storage [28]. In addition, it is important that during storage moisture content be minimized as much as possible to prevent microbial attack, enzymatic and hydrolytic degradation. *Chrysophyllum albidum* gum has fairly high moisture content suggesting that it is slightly hygroscopic in nature and should be stored in air tight containers. Powders are required to flow and the efficacy with which they do so is

dependent on both process design and particle properties. Hausner's ratio and the angle of repose have been used to describe the flow properties of solids and they are related to the interparticulate friction or resistance to movement between particles [29]. Hausner's ratio above 1.25 suggests poor flow, Carr's Index lower than 16 % indicates good flowability while values beyond 35 % indicate cohesiveness. The angle of repose and Carr's index of *Chrysophyllum albidum* gum are high (Table 1) and indicative of a slightly poor flow. However, it should be noted that the angle of repose is not an intrinsic property of the powder and it is largely dependent on the method used. However, the addition of a glidant or adjustment of processing conditions can enhance the flowability of the gum.

FTIR. FTIR is a technique used to examine the formation of intra and inter molecular hydrogen bonds in the gum, the correlation between the nature of hydrogen bond and the physical and mechanical properties of the gums. The wide band at 3445 cm^{-1} is attributable to hydrogen bonded OH groups that contribute to the complex stretches associated with free inter and intra molecular bound hydroxyl groups which make up the gross structure of carbohydrates [30]. Peaks at 1638 and 1454 cm^{-1} corresponds to C=C stretching inside the benzene ring while the wide stretch in the region between 2000-2700 cm^{-1} suggest 0=C=H band stretching. Studies have shown that natural gums are weakly anionic due to the presence of sugar acid fractions [31]. Absorption peaks between 1638 and 1454 cm^{-1} could be indicative of carboxylic groups of the galacturonic acid residues. The absorption bands between 800 and 1200 cm^{-1} represent the fingerprint region for carbohydrates [32]. All these are in conformity with a polysaccharide structure.

Morphology. The size and granule shape of a pharmaceutical material is to an extent determined by the botanical source. Therefore, these characteristics in addition to assisting in differentiating between various materials, also give an indication of processing parameters. Figure 3 shows the scanning electron microscopic image of *Chrysophyllum albidum* gum powder. The SEM images reveal that the particles are round and regular in shape. The procedure of extraction as well as the method of purification of the product may greatly affect the surface structure of the gum [33]. The low enthalpy (Table 2) can be attributed to the presence of a more regular or small and oval particles [34]. This is consistent with the SEM images. The offset and peak temperatures were high. The small endothermic peaks at 180 and 254 ^0C might be due to breaking of bonds and release of energy.

Compressional characteristics. The compressional characteristics were evaluated using density measurements and indices of plasticity from Heckel and Kawakita plots. Table 3 shows that D_B which describes the re-arrangement phase at the early stages of compression is higher in corn starch. The value of 'a' which describes the extent of consolidation as the powder becomes closely packed is smaller in *Chrysophyllum albidum* gum. Smaller values of the constant 'a' indicates good packing even without tapping which suggests that that *Chrysophyllum albidum* gum exhibit good packing than corn starch. The mean yield pressure, P_y gives an indication of direct compression and it is a reflection of the propensity of the material to deform plastically or by fragmentation. Low values indicate easy compression, good densification, low resistance to pressure and fast onset of plastic deformation [35]. The P_k in Kawakita plot is the pressure required to produce 50 % volume reduction of the powder bed [23]. Low value indicates that the material is soft, readily deforms plastically and exhibits a high amount of overall plastic deformation. Table 3 indicates that *Chrysophyllum albidum* gum not only showed

a slower onset of plastic deformation than corn starch but had a lower overall amount of plastic deformation during compression process. The addition of a plastic material to *Chrysophyllum albidum* gum may be necessary when compressed on a tablet press.

Conclusion. Overall, this study established the fundamental characteristic of *Chrysophyllum albidum* gum and explains the rationale behind its use as raw materials in pharmaceutical formulations.

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