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Research Article

Characterization of *Khaya Senegalensis* Gum: Effects of Drying Methods on the Physicochemical Properties

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ABSTRACT

The study was aimed to assess the effect of drying methods on physicochemical properties of *Khaya senegalensis* gum. Gum exudates obtained from the bark of *Khaya senegalensis* plant was extracted and subjected to freeze drying and vacuum oven drying. Gel permeation chromatography, Elemental analysis, attenuated total reflectance Fourier transforms Infra red spectroscopy were used to characterize the gum. The effect of drying methods on the gum samples were assessed based on Differential scanning calorimetry, Scanning electron microscopy, Dynamic vapour sorption, particle size analysis via laser diffraction techniques, colour and density measurements. An average molecular weight of 60 KDa expressed as the Dextran equivalent and a polydispersity of 2.17 with a retention time of 18,485 min was recorded for the vacuum dried gum. The vacuum dried gum occurred as irregular discreet coarse particles, containing both amorphous and crystalline portions due to a glass transition of 65° and melting temperature of 182.35°C. It also displayed excellent flow and compressibility properties whereas the freeze-dried gum appeared flaky, porous with large surface area. The mechanism of water sorption was found to be initial surface adsorption and subsequently bulk absorption for both freeze dried and vacuum dried gum samples. The coarseness, water sorption and retention ability of the vacuum dried gum provides a potential for its use as a disintegrant in solid dosage formulations whereas the freeze-dried gum will be suitable for use as a binding agent due to its smaller particle sizes that enhanced solubility. Furthermore, the presence of nitrogen in *Khaya senegalensis* gum structure explains its partial solubility and the azo aromatic group suggests biodegradability and candidature for drug delivery to the colon.

Keywords: Khaya senegalensis Gum; Average Molecular Weight; Amorphous; Crystalline; Direct Compression Excipient; Disintegrant 2 Freeze and vacuum dried Khaya senegalensis gum characterization

INTRODUCTION

Natural gums are plant derived polymers that are characterized by swelling and high viscosity in water or aqueous solutions. They are non-toxic, non-irritant, biocompatible, inexpensive, abundant, available, biodegradable and easily undergo chemical reaction[1,2]. This plant derived polymers have been useful in pharmaceutical applications and have been well implicated in transforming drug delivery due to their appealing and derivatizable characteristics[3–5]. *Khaya senegalensis* gum (KSG) is a plant gum that exudes in a molten form from the bark of *Khaya senegalensis* plant after an incision or as a result of injury, and then dries to form long, thin glass-like translucent fragments. A number of research works have been carried out to assess the potentials of Khaya gum in pharmaceutical formulations. For example, binding properties of KSG were found to be similar to that of a higher concentration of acacia[6]. The use of Khaya and Albizia gums as compression coatings for colon specific drug delivery systems were investigated. The findings showed that both gums were capable of protecting the drug from the[7] simulated environment of the stomach and small intestines. Comparing the binding effect of gums from *Khaya senegalensis* and *Khaya grandifolia*,[8] reported that KSG showed better tablet binding property, mechanical strength and long dissolution and disintegration times with increased gum concentrations. The properties of KSG in liquid preparations were also assessed[9]. The authors observed that KSG displayed high

viscosity values in water with increasing concentration and an appreciable swelling power that may provide potential for use as modified release material and further reported its gradual release profile from a suspension[10,11]. Like many natural polymers with variable functionality, its use as a binder, disintegrant and also in modifying drug release in combination with other polymers in tablet formulations was studied[12,13]. These works however revealed its potential as having many pharmaceutical applications albeit only sparsely characterized[9,14,15]. A more detailed understanding of the physicochemical characteristics of the gum will help to understand and guarantee the behaviour of the material during formulation phases of drug development[16]. This research thus, focuses on an in-depth characterization of KSG using more recent techniques. The physicochemical and functional properties of natural plant-based biopolymers are extensively influenced by many factors such as the chemical composition and molecular structure which can be affected by the extraction, purification, drying or other modification processes[17]. Drying process have been shown to have a significant effect on the physicochemical properties, functional and formulation characteristics of materials. Spray drying, freeze drying, vacuum oven drying among others are most commonly used methods of drying plant gums[16,18,19]. The aim of this study is thus, to characterize *Khaya senegalensis* gum using recent analytical techniques such as Gel permeation chromatography (GPC), Attenuated total reflectance Fourier transform infrared spectroscopy (ATR- FTIR) and CHN for elemental analysis and then assess the effect of vacuum oven and freeze drying on the physicochemical properties of KSG with focus on particle size, surface morphology, thermal properties, dynamic vapour sorption (DVS) and density measurements to determine appropriate functionality offered by the different drying processes employed.

MATERIALS AND METHOD

Materials

Crude *Khaya senegalensis* gum obtained from *Khaya senegalensis* trees, Purified *Khaya senegalensis* gum (KSG), Ethanol 97% denatured with 3% IPA, Disolol® (Chem.-Lab NV, Belgium), Potassium sorbate Ph. Eur (SA Aca Pharma NV). All other materials were of analytical grade.

Collection, Authentication and extraction

Gum exudates were obtained dry from *Khaya senegalensis* trees grown in Zaria town of Kaduna State. The crude gum exudate was authenticated in the herbarium unit of the Department of Biological Sciences of Ahmadu Bello University Zaria where a voucher number 900181 was assigned and specimens were kept. The crude KSG was coarsely size reduced and a 500 g quantity was weighed on a Metler Toledo balance (AB 204, Switzerland) and dispersed whilst stirring intermittently in 5L of deionised water, preserved with 0.1% potassium sorbate and kept at room temperature for 24 h to allow for complete hydration. The dispersion was then homogenised with a Silverson mixer (model L4S-R, Switzerland) and 15 L of deionised water was further added to obtain an even dispersion which was stored at 6ºC for 24 h. Thereafter, a total of 12.5 L of ethanol was used to precipitate the gum from the aqueous dispersion. At this point two layers formed and particulates settled at the bottom, settling was allowed for 30 min after which KSG was filtered through Whatman filter paper no.1 and then spread on absorbent paper prior to drying in a vacuum oven (WTC Binder, Tuttlingen, Germany) at 40ºC for 48 h, the gum obtained was referred to as KSG-VD (vacuum dried). The gum was again extracted as described above and then dried in a freeze dryer (Telstar Bomba RD-18, Spain). The freeze-drying cycle was operated at shelve temperature - 5°C for 25 min then freezing at - 45º C for 150 min and there after drying at a low vacuum pressure of 0.100 mbar for 30 min at - 15º C for 13 h 30 min and then at 0ºC for 15 min and finally at 10ºC for 9 h. The resulting gum from this drying process was labelled *Khaya senegalensis* gum Freeze dried (KSG- FD).

Percentage yield of the purified gum

The crude (w1) and the dried, precipitated and purified gum (w2) samples were weighed and the percentage yields were computed as given in the equation below. The colour, odour and texture were observed physically.

$$\% \text{ yield } = w1/w\,2 \times 100 \quad (1)$$

pH and aqueous solubility determination

A 1.0% w/v concentration of KSG-VD and KSG–FD were separately prepared in deionized water and the pH determined using a pH meter. Aqueous solubility of the gum samples was determined by gravimetric analysis as described by Nep and Conway[20].

Elemental analysis

The method of CHN with calibration based on K factors was employed, a 2.361g of KSG was used for this experiment. The percent composition of Carbon, Hydrogen and Nitrogen in the sample were determined using Eager 300 elemental analyzer.

Thermal analysis

A 5mg quantity of KSG-VD and KSG- FD were individually encapsulated in hermetic pans and covered with hermetic lids (A39817-03/ USA) in order to measure their degradation, glass transition (Tg) temperatures and melting points (Tm)using Differential scanning calorimeter (DSC Q2000 V24.10 Build 122) connected to a Universal V4.5A TA instrument software. A heating rate of 10° C/ min was used

and ramping was performed from-20 °C to 250 °C using nitrogen as purge gas[21].

Dynamic vapour sorption

A 4.74 g quantity of KSG-VD was placed on the DVS sample pan of an ultra-microbalance with a mass resolution of ± 0.1μg and dried under a stream of dry Nitrogen gas at 21°C. Humidity was increased in 10% RH steps to 100% RH for the sorption phase and then decreased in a similar fashion for desorption phase. This same procedure was performed on KSG-FD powder (3.29 g). The nature of the gum after drying led to the variation in weights of gum sample used for the experiment.

Determination of relative molecular weights by means of Gel Permeation Chromatography (GPC)

The conditions for GPC experiment were; Pump: 610 fluid units (Waters) + 600 Controller (Waters) Column setup: Guard column SB-G (Shodex) + 2x Ohpak SB806M-OH (Shodex) and a Detector: 410 RI detector (Waters) set at a flow rate of 1ml /min. The column and detector temperatures were set at 80°C and 50°C respectively. For the purpose of SEC, a 5.0 g quantity of KSG-VD was dispersed in distilled water to dissolve. The upper soluble part of this dispersion was pipetted onto petri dishes and then freeze dried as previously describe above in order to get a purer form of the gum. A 10 mg sample of gum was separately dissolved in 2 ml phosphate buffer solution (pH 7) and allowed to stand for 1h, then conditioned in warm water bath at 50°C for 30 min and further sonicated for 15 min to ensure complete dissolution. The solution was filtered using a syringe filter 0.45μm in size before injection into the GPC system (only a partial filtration of the gum dispersion was achieved). The retention time (RT), number average molecular weight (Mn), weight average molecular weight (Mw) and polydispersity index (PI) were determined.

Attenuated Total Reflectance Fourier Transforms Infra- Red Spectroscopy (ATR- FTIR)

This experiment was performed on purified KSG-VD solid sample as described by[21] with slight modification. Spectra were recorded using an ATR-FTIR spectrometer (Thermo Fisher Scientific, Nicolet iS5 ATR-FTIR spectrometer, Erembodegem-Aalst, Belgium) was repeated twice. A diamond ATR crystal was pressed against the sample and the spectra were collected in the range 4000 - 550 cm-1 range with a resolution of 4 cm-1 and averaged over 128 scans. An identity to the polysaccharide depending on its chemical structure and chain conformation was determined.

Surface morphology

Images of the surface appearances of the gum samples were viewed at x100 and x1000 magnifications and recorded using a Scanning electron microscope Quanta 200F (FEI, Eindhoven, The Netherlands) operated at an acceleration voltage of 20 kV.

Particle size distribution by Laser diffraction

The unmilled KSG powders were directly fed into the dispersion funnel and the optical concentration was maintained at a range 5.0-8.3%. The average median particle size (D50), the specific surface area and span for KSG powders were determined using a laser diffraction (Master sizer, Malvern, Worcestershire, UK) at a feed rate of 3.0 g and jet pressure of 1.3 bars. The mean value of three determinations was used to plot a size distribution curve.

Density measurements

The bulk volume (V0) of 30 g of gum samples were recorded in a 100 ml measuring cylinder as well as the volumes after 500 (V500) and 1250 (V1250) taps in an automated tapping machine (J. Englesman, Ludwigshafen, Germany). The experiment was performed in triplicate according to USP specifications[22]. The bulk and tapped densities, Hausner ratio and Compressibility index were calculated as given below;

$$\text{Bulkdensity}\,(B\rho) = \text{mass of powder (g)}/\,\text{bulk volume (ml)} \quad (2)$$

$$\text{Tappeddensity}\,(T\rho) = \text{weight of powder (g)}/\,\text{tapped volume (ml)}$$

where tapped volume at V500 and V1250 were computed separately for the tapped volumes at V500 and V1250.

$$\text{Hausner's ratio} = \text{tapped density} \,/\, \text{bulk density} \quad (4)$$

$$\text{Carr}\,'\text{s index} = \frac{\text{Tapped density-Bulk density}}{\text{Tapped density}} X100 \quad (5)$$

RESULTS AND DISCUSSION

Effect of drying methods

The physicochemical properties of the gums obtained either by vacuum or freeze drying are summarized in Table 1 .

The yield of KSG was unaffected by either of the drying techniques employed. The colour of gum powder was affected by the drying methods. Vacuum drying produced gum powder with a brown colour while the freeze-dried gum appeared cream coloured with more aesthetic quality which can be applicable where formulation is not required to be coloured.

Long resident time and temperature differences are two causes in producing dark and red coloured flax seed

Table 1: Physicochemical Properties of Purified KSG-VD and KSG-FD Powders

Parameters	KSG- VD	KSG-FD
Colour of crude	Brownish red	Brownish red
Colour of purified	Light brown	Cream
Taste	acidic	acidic
pH (21°C)	4.55	4.62
Texture	gritty	fluffy
Percentage yield (% w/w)	50	50
Aqueous solubility (% w/v)	15	16.3
Moisture content (%)	4.05 ±0.24	8.35 ± 0.10
particle size (μm)		
D10	67.0 ±3.93	19.57±2.35
D50	184 ±10.1	105.2 ± 20.8
D90	395.49 ±17.51	380.62 ± 48.4
Span	1.88 ± 0.07	3.46 ± 0.23
Specific surface area (m2/g)	0.058±0.00	0.165 ± 0.04
Bulk density (g/cm-3)	0.811 ±0.0	0.215 ± 0.0
Tapped density (g/cm-3)	0.963 ±0.01	0.286 ± 0.01
Hausner ratio	1.18 ± 0.01	1.33 ± 0.04
Carr's index (%)	15.76 ± 0.78	24.85 ± 2.57

gum powder[19]. The change in colour of KSG-VD can be attributed to temperature differences of the drying methods. Freeze drying imparts light weight, increases stability, surface area and solubility as well as porosity to materials subjected to the process[23–25] .These effects were observed as the drying method was changed from vacuum to freeze drying. A similar occurrence on plant gums experimented have been reported[14,16]. The pH of KSG in this work was found to be mildly acidic. This is in agreement with previous works[9,15]. Although a slight shift in pH increased solubility, a decrease in particle size increased the effective surface area of the gum particles in contact with aqueous milieu, this increased solubility and ionization of ionizable components of the excipient in water. The solubility of drug is often intrinsically related to drug particle size; as a particle becomes smaller, the surface area to volume ratio increases. The larger surface area allows greater interaction with the solvent which causes an increase insolubility[26]. Basic excipients promote oxidation of susceptible drugs when used for their formulations[15]. Therefore, acidic and neutral hydrocolloids are more widely used for pharmaceutical formulations[9]. A significantly higher absorption was observed for the more amorphous of the two gums, KSG-FD changing faster from glassy to rubbery state. The moisture sorption kinetic profile showed the highest mass absorption (% change in mass) at RH 98 for both gum powders. The mass increase been 44.66% for KSG-VD and the most absorbed 57.93% KSG- FD. The mechanism of moisture uptake can be explained as initial surface adsorption at low RH and subsequently bulk absorption when exposed over a long period of time at RH 70%. Although, the measurement parameters for both samples was the same (2 cycles of 0-98 % RH, water). Water caused the amorphous content to crystallize at RH 30 point during desorption (cycle 2) due to change in crystal lattice (Figure 1) whereas an early recrystallization (RH 98, cycle 1) was observed for KSG-FD due to its hygroscopic nature (Figure 2). It is presumed that water in amorphous material is absorbed which therefore results in a higher water uptake and the high affinity for water may be as a result of instability of the amorphous content[27,28]. A gradual increase in water uptake created a hysteresis that remained throughout RH 0 to 98% (Figure 3). This is a typical hysteresis shape for many pharmaceutical and food materials. An obvious characteristic is that the gap remains over the entire partial pressure range. The hysteresis was between 0 to 90% RH in Figure 4, it revealed a good agreement between the adsorption and desorption branch at 90-98% RH with no significant hysteresis because the second cycle was identical to the first cycles at this point confirming that there was no irreversible effect.

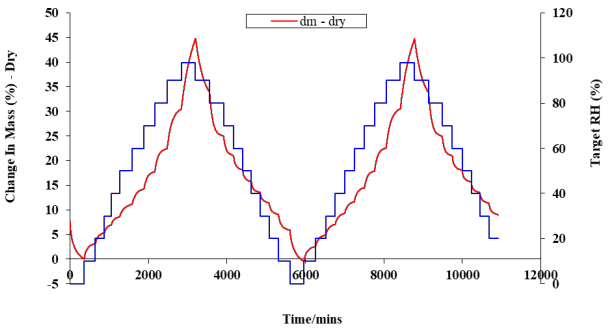

Fig. 1: Water sorption kinetics for *Khaya senegalensis* gum vacuum dried (KSG-VD) at 21 °C

Hysteresis is the gap or difference between adsorption and desorption isotherms in cases where they do not meet[29]. Partially amorphous materials tend to exhibit considerable bulk absorption of water which is a slow, diffusion-controlled process and therefore non-equilibrium regime[30]. The high percent moisture content of KSG-FD was expected due to increase solubility and porosity imparted by its drying method. However, this value still remained lower than the USP 2007 specification of 15% w/w for natural gums. Other authors,[16,17,19] also noticed that freeze drying increased solubility as well as hygroscopicity on gums. Nonetheless, whichever method is employed in drying KSG, it is required to be stored in air tight containers to improve stability during storage and economic importance. The particle size distribution (PSD) curves of the gum samples (Figure 5)

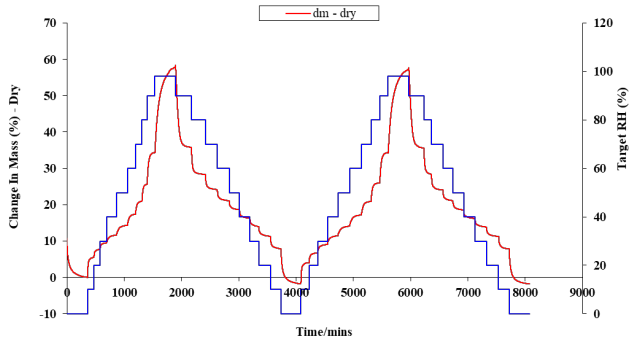

Fig. 2: Watersorption kinetics for *Khaya senegalensisgum* freeze dried (KSG-FD) at 21 °C

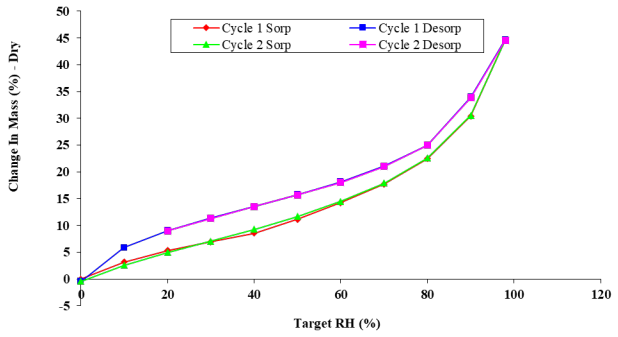

Fig. 3: Moisture sorption-desorption Isotherm for *Khaya senegalensis* gum Vacuum dried (KSG-VD) at 21 °C.

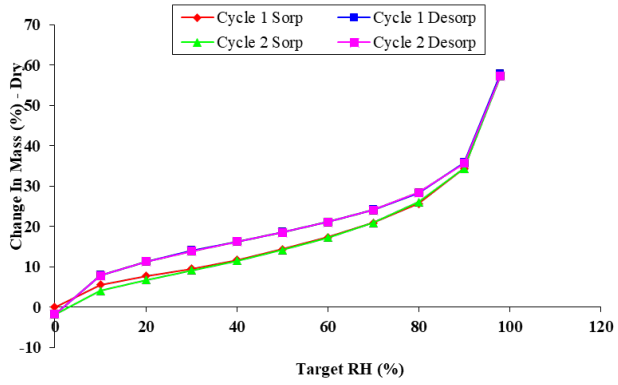

Fig. 4: Moisture sorption-desorption Isotherm for *Khaya senegalensis* gum freeze dried (KSG-FD) at 21 °C

appeared positively skewed that is, the tail elongated towards the larger particles and therefore not a normal distribution where the central values (mean, median and mode) is equal.

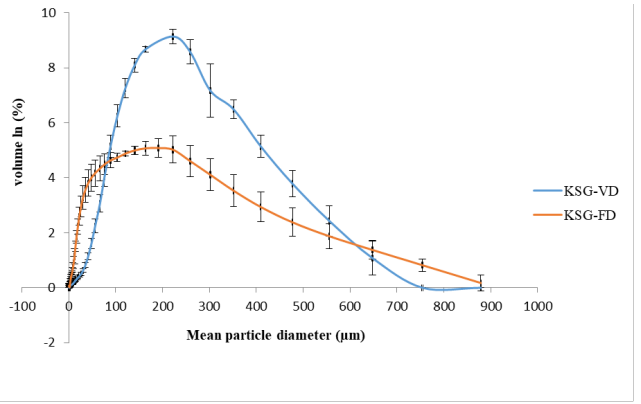

Fig. 5: Particle size distribution curves of unmilled *Khaya senegalensis* gum vacuum dried (KSG- VD) and *Khaya senegalensis* gum freeze dried (KSG- FD) powders

The D10 and D90 values of the PSD curve showed that 10% of the particle population of KSG-VD are composed of fine and 90% of the population consisted of coarse particles. Similarly, D90 values of KSG-FD revealed 90% of particles are below 380.6 $\pm$ 48.3 μm and 10% (D10) of the population comprised of particles $\leq 20\mu$m in diameter. The median particle diameter D50, revealed half of the particle population of KSG-FD to be much smaller than that obtained with KSG-VD (Table 1,Figure 5). During the extraction of KSG, two portions resulted, a fine soluble and a coarse insoluble layer. This large particles in KSG-FD are the insoluble coarse portion of the gum which was unaffected by freeze drying while increase in surface area was the result of the effect of freeze drying on the soluble particles. The coefficient of variation (COV) for the three replicate determinations of D10 and D90 were $\leq$ 15 % for the both gums dried by vacuum and freezing while D50 for KSG-VD was within the 10 % USP <429> specification, indicating excellent method repeatability, that of KSG-FD failed the requirement.

The SEM images (Figure 6) revealed differences in shape and crystal habits in the surface features of KSG-VD and KSG-FD powders and defined clearly the fineness and coarseness of the gums by a distinct difference in particle size and shape. The SEM images of KSG-VD particles revealed particles larger than 100 μm, almost twice the size estimate for 50% of the powder population of KSG-FD. An increase in specific surface area (SSA) of KSG-FD was noticed with the appearance of flaky irregular small particles in contrast to a more defined rectangular multifaced discreet particles of KSG-VD agreeing with its gritty texture ($p<0.05$), (Table 1). This implies that the SEM images represented well the data obtained via laser diffraction technique. Works performed were able to correlate data

obtained from laser diffraction and SEM images of different grades of microcrystalline cellulose wherein fine and coarse particles were well estimated by both physical techniques and supported one another[31]. The effects of drying methods on textural characteristics of gums was also reported[32].

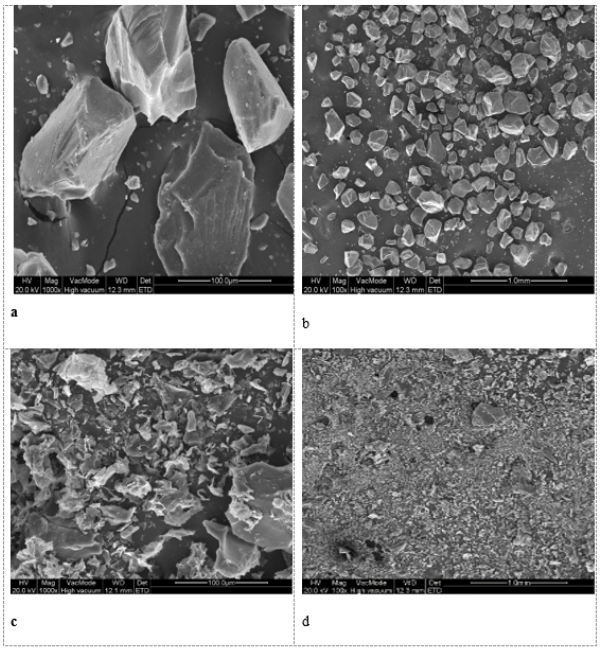

Fig. 6: Scanning electron micrograph (SEM) images of **a & b** *Khaya senegalensis* gum vacuum dried (KSG- VD) and **c & d** *Khaya senegalensis* gum freeze dried (KSG- FD) x1000 and x100 magnifications

Influence of drying process on powder flow and compressibility

The bulk density of a powder is primarily dependent on particle size, particle size distribution and particle shape[33]. The density of gum powders was affected by the drying methods employed. The bulk and tapped densities of KSG-VD were higher than values obtained for KSG-FD ($p<0.05$) (Table 1). This implies that KSG-VD is denser and more consolidated and thus, will occupy less space during packing. When poured into cylinder coarser particles of KSG-VD were more consolidated and had excellent free flow compared to fines and flaky particles of KSG-FD powder that had poor flow as a result of the drying method. The values of Hausner ratio and Compressibility index further confirmed this observation. The Compressibility index and Hausner ratio are measures of the propensity of a powder to be compressed. As such, they are measures of the powder ability to settle and they permit an assessment of the relative importance of interparticulate interactions[34]. According to Hausner, powders with low interparticle friction, such as coarse spheres, have ratios of approximately 1.2, whereas more cohesive, less free-flowing powders such as flakes have ratios greater than 1.6. Similarly, powders with 12-16% compressibility index are said to have good free flowing property whereas powders with Carr's index of 23-28% are very fluid with poor flow properties[35]. Again, the flow properties of these gum powders agree with the SEM and PSD results.

Elemental analysis

Carbon 37.83 %, Hydrogen 5.74 % and Nitrogen 0.85 % were elemental composition found to be present in KSG-VD. The nitrogen content indicated the presence of amino acids which has been found in plant gums in small and relatively large quantities ranging from 0.04 % in *Acacia leucoclada* to 5.6 % in *Azadirachta indica* gum. A nitrogen content of 0.56 % was reported in *Khaya grandifolia* gum, another species of Khaya[36–38]. The higher the nitrogen content in the plant gum the more insoluble gel it forms in aqueous dispersions[39]. The partial insolubility of KSG may also be attributed to the nitrogen content found in this gum. Other elements including magnesium, sodium, calcium, copper (II) ions and a high content of potassium ions were reported to be present in KSG[15].

Differential thermal analysis

Partially crystalline polymers give rise to very broad melting peaks because of the size distributionof the crystallites which can melt over a wide range of temperature[40,41]. The thermal curves were characterized by broadness and this was more pronounced in the DSC curve of KSG-FD (Figure 7), although sealed in a hermetic pan and lid, it melted due to bound moisture acquired from its drying process and showed an endothermic peak at 43.54°C and 196.30°C related to loss of moisture or dehydration and a somewhat melting peak[42,43] In (Figure 8), the endotherm is comparatively less broad than that of KSG-FD (Figure 7). A glass transition (Tg) marked at 56.14°C and a melting point at 182.35°C with no evidence of degradation at the end of the transition was revealed. The Tg indicates the presence of amorphous content while the characteristic melting curve depicts crystallinity. Melting points are increasingly sharper with smaller sample sizes and purer substances due to crystalline rearrangement, fusion or solid-state transition. Impure substances often show several peaks[41] and such peaks were not seen in all the curves obtained for both KSG-VD and KSG-FD powders and could be an indication for a level of purification in the extraction stage. The gum powder will remain stable at room temperatures and above due to the high Tg. The slight differences in Tg and Tm of the KSG-VD and KSG-FD gum samples suggest structural and functional group differences in the samples, an effected of the drying methods. It should be noted that this is the first time DSC

scans of KSG obtained by two drying methods are displayed.

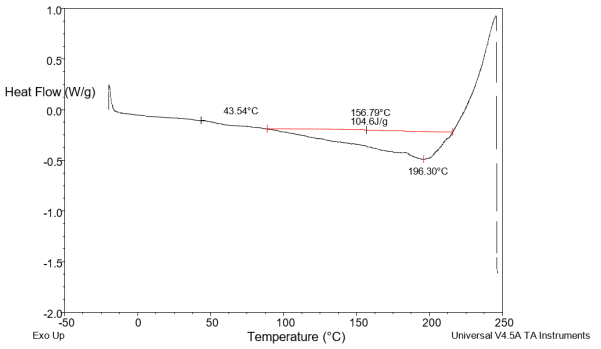

Fig. 7: Differential scanning calorimeter (DSC) thermogram of KSG-FD

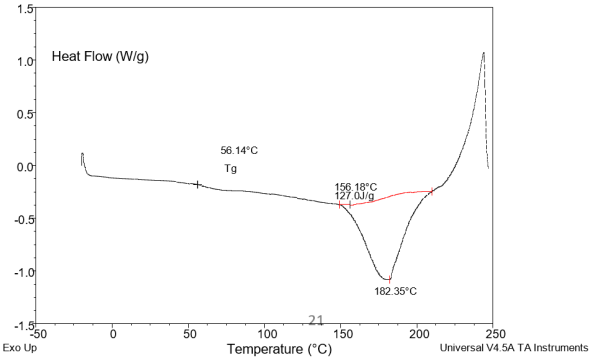

Fig. 8: Differential scanning calorimeter (DSC) thermogram of KSG-VD

ATR- FTIR spectra of KSG-VD

The possible functional groups associated with band widths of ATR-FTIR spectra of purified KSG-VD (Figure 9) are shown in Table 2 . The presence of carboxylic acids supports the assumption of CO2 evolution as a mechanism of thermal transition of gums in the DSC thermograms[44]. Similarly, the presence of carboxylic acid at 1726 cm-1 frequency in cashew gum was an indication of its presence in cellulose containing compounds[45]. The results of ATR-FTIR corrobrated with elemental analysis as both showed the presence of amide groups. Further, the possible presence of Azo (-N=N-) groups at 1416 cm-1in KSG-VD suggests that they can be degraded in the small intestine where the enzyme diazo reductase is present and can be considered biodegradable[4,46]. This implies that KSG-VD can be useful in modified release especially in controlling drug release along the GI segments. In relation to sugar components of the gum, the hydroxyl, carbonyl and methyl functional groups represent the backbone structure of carbohydrates while C-O stretch at 1239 cm-1 represent aromatic compounds of rhamnose, galactose and galacturonic acid and the medium peak at 1725 cm-1 due to C=O may be related to galacturonic acid. Galacturonic acid was reported as a constituent of *Khaya grandifolia* gum[47–49].

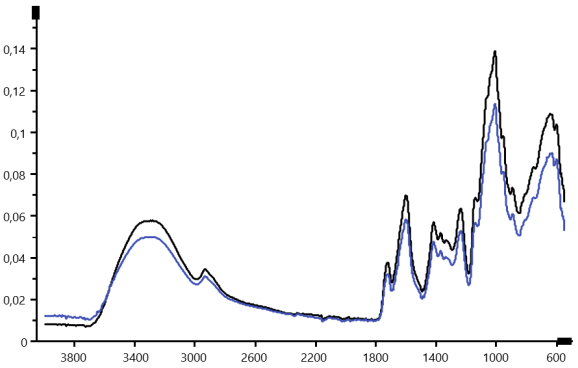

Fig. 9: Attenuated total reflectance Fourier transforms infrared (ATR FT- IR) spectra of KSG-VD

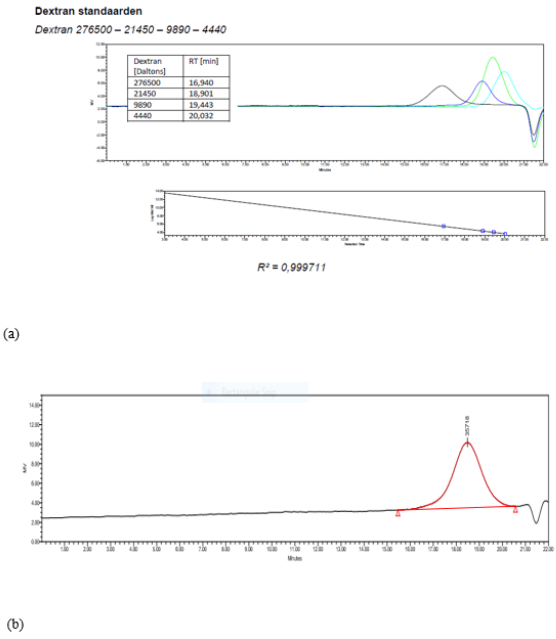

Fig. 10: (a) Retention times for Calibrated Standard Dextran and (b) Retention time of highest peak of *Khaya senegalensis* gum via Gel Permeation Chromatography

Gel Permeatio n Chromatography (GPC)

Polymers are composed of repeated units of monomers chemically bonded together to form a long chain. Knowledge of the chain length is used in understanding their physical

Table 2: Interpretation of possible functional groups associated with band widths of ATR-FTIR spectra of purified KSG-VD

Bandwidth frequency	Possible functional group
1725 cm-1	C=O: stretch, different types: saturated aliphatic ketones, saturated aliphatic aldehydes, aryl esters, formates, sat aliphatic carboxylic acid dimers
1600 cm-1	N-H: primary amide, primary amine NH2+: amine salt CO2-: asymmetric stretch, carboxylic acid salts NH3+: amine salt (C=C: aromatic structure)
1416 cm-1	N=N: aromatic azo compound C-N: primary amide, amide III band C-O stretch and OH carboxylic acids O-H: primary, secondary alcohols
1239 cm-1	C-O: carboxylic acid dimers N-H: secondary amides, amide III C-O-C: saturated aliphatic esters C-O: acetic acid ester CH3COOR Cyclobutanes R-O-Ar (alkylthicketones)
1100-950 cm-1	Thioamides N-C=S CH3-C: aliphatic CH3-Ar Saturated aliphatic ethers
633 cm-1	N-H primary amides C=C-H bending, terminal acetylene groups N-C=O bending, primary saturated aliphatic amides, NO2, nitroalkane, Vinyl compounds

Table 3: Average molecular weight values for *Khaya senegalensis* gum

Parameters	RT (min)	Mn	Mw	Mp	Polydispersity
Values (Da)	18,485	27,494	59,536	35718	2.17

Key: RT = retention time, Mn = number average molecular weight, Mw = weight average molecular weight, Mp= molecular weight of highest peak

properties such as mechanical strength, solubility and brittleness. Chain length is usually expressed in relation to relative molecular mass of monomers and the number of monomers in a chain. Although all synthetic polymers polydisperse, they have unequal chain length and therefore the molecular weight is not a single value and has to be expressed as an average[50]. Natural polymers on the other hand are often monodisperse i.e. they have molecules of uniform mass in a single chain[51,52]. A monodisperse curve with a peak molecular weight (Mp) of 36K Daltons due to Dextran as a calibration material was obtained for KSG (Figure 10 ,Table 3). The broadness of a molecular weight distribution of a polymer is expressed by its polydispersity index (Mw/Mn)is important to any research on cellulose and its derivatives because they influence dissolution, regeneration and accessibility or reactivity of the biopolymer[53]. A dispersity value of 2.17 is indicative of step polymerization reaction. Polymerization usually occurs as a reaction between two functional groups e.g. carboxyl and hydroxyl groups while a step polymerization reaction involves a reaction between monomers with two or more functional groups. This finding supports the ATR-FTIR results with several functional groups present at an absorption peak. The peak molecular weight (Mp) is quoted for very narrowly distributed polymers such as those used for standard calibration. The Mp of 36 K Dalton and the retention time obtained in this experiment for KSG indicates that it composes of a Dextran -like polymer. The presence of Dextran-like material may be an indication for the non-gelling property of KSG and also its good disintegrant action that has been earlier mentioned. Polysaccharides as a group have wide range of properties from insoluble (cellulose) to soluble and high swellable (guar, starch); low viscous to highly viscous (acacia and guar gum), gelling agar to non-gelling dextran[54] To this end, KSG standing alone cannot be used to formulate extended release formulations but may act as a release modifier to hasten drug release in combination with other polymers.

CONCLUSION

The physicochemical properties of *Khaya senegalensis* gum obtained via vacuum and freeze-drying methods have been examined. The drying method was found to impact the physical properties of the gum like colour, texture, moisture sorption, particle size and surface morphology as well as the thermal properties. The pH and gum solubility in water were only slightly affected. A high glass transition and more distinct melting curve of the vacuum dried gum depict stability at room temperature. The freeze-dried gum particles were fluffy in texture, smaller in diameter, more hygroscopic and dried faster compared to the vacuum dried gum that was coarse, larger in size and took up less moisture but retained it for a longer period of time. The mechanism of water sorption was found to be initial surface adsorption and subsequently bulk absorption for both gum samples. The coarseness, water sorption and retention ability of the vacuum dried gum provides a potential for its use as a disintegrant in solid dosage formulations whereas the enhanced solubility due to smaller particle size of the freeze-dried gum will allow for its use as a binding agent. The retention time (RT) and the peak molecular weight (Mp) of the vacuum dried gum revealed its possession of Dextran-like components however; more research work can be performed for further confirmation.

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