

Extraction and Physicochemical Characterization of *Adansonia digitata* L. Mucilage

H.S. MAHMUD^{1,2} A,B,C,D,E, R.A. OYI^{2A,C,E}*, T. S. ALLAGH^{2A,E}, Y.K.E. IBRAHIM^{3A,C,E,F}

¹Laboratory of Pharmaceutical Technology, Ghent University, Ghent, Belgium

²Department of Pharmaceutics and Industrial Pharmacy, Ahmadu Bello University, Zaria, Nigeria

³Department of Pharmaceutical Microbiology and Biotechnology, Ahmadu Bello University, Zaria, Nigeria

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 article.

Abstract

Background: *Adansonia digitata* leaves are edible and they contain a significant amount of mucilage that has shown potentials for pharmaceutical application. However, there is paucity of information on this mucilage contained in its leaves.

Objective: This study aimed to determine a suitable method for extraction of *Adansonia digitata* mucilage (ADM) as well as to characterize its physicochemical properties.

Materials and Methods: ADM was extracted from the aqueous dispersion of the dried leaves powder of *Adansonia digitata* L. in water via precipitation with ethanol. Physicochemical characterization of ADM was based on viscosity, chemical composition, particle size characterization via QICPIC, Scanning Electron Microscopy (SEM), Attenuated Total Reflectance Fourier Transforms Infrared (ATR -FTIR) spectroscopy, Nuclear magnetic resonance (NMR), moisture content and Dynamic vapor sorption (DVS) studies as well as pH and aqueous solubility tests. In addition to this, colour of the mucilage and thermal analysis via DSC were used as criteria for selection of the most suitable method for extraction of ADM.

Results: Colour and thermal analysis revealed a level of purity in the extraction process. The irregular needle like structures of ADM revealed by SEM was found to be mildly acidic with a high viscosity that is concentration dependent in aqueous medium. Thermal characterization revealed a glass transition (T_g) and melting temperatures (T_m) of 55.61 °C and 179.10 °C respectively. Finger prints of functional groups revealed azo aromatic groups and other chemical constituents of sugars including glucose, galactose, arabinose and rhamnose, and sugar acid form of galacturonic acid were identified by NMR. Moisture sorption provided insight into water sorption mechanism, processing, packaging and storage conditions of the mucilage.

Conclusion: Extraction of ADM via a heat free method improved its colour and purity based on the technique used. The mucilage was found to be a highly viscous and hydrates rapidly with good gelling properties. In addition its moisture sorption characteristics and glass transition temperature gave insight into its stability, storage and packaging conditions. These properties reveal its potential for many pharmaceutical applications including binding, coating, gelling agents as well as matrix forming.

Keywords: *Adansonia digitata* mucilage, Extraction, Glass transition, Sugars acids, Needle like shape, Viscosity

INTRODUCTION

Like many other developing countries, Nigeria relies on importation of medicinal agents and raw materials (Nep & Conway, 2010b) including synthetic polymers for use in drug or food processing. Although plant producing polymeric materials are available in Nigeria, many are used only as food, some exported unprocessed and several have remained unexplored and yet to be fully discovered for their pharmaceutical potential. An example of this is *Adansonia digitata* L. (family *Malvaceae*). The plant *Adansonia digitata* L. (Baobab) is majestic and is the largest succulent in the world with a height of 23 m and 10-12 m in diameter (Wickens, 1982; Chadare *et al.*, 2008). It is found in the hot dry savannahs of sub-Saharan Africa and also grows in populated regions as a result of secondary cultivation. The tree is known to have a life span of several hundred years and it is resistant to fire (Zahra'u *et al.*, 2014). *Adansonia digitata* ("Baobab" in English, "Kuka" in Hausa and "Igiöse" in Yoruba) is found to be among the most effective plants that prevent water loss. Though not widely cultivated, it has been used by humans for multiple purposes such as food or medicine (Gebauer *et al.*, 2002). Due to its traditional application in cosmetic, nutrition and medicine, the plant fruit pulp has been approved for importation into the EU (Buchmann *et al.*, 2010). Carbohydrate is said to be the chief component of the leaves with about 60-70 % w/w followed by protein 13-15 %, then 4-6 % of fat, 11 % fibre and 16 % ash. In addition, 7 to 10 % of the dry matter of leaves is mucilage (Woolfe *et al.*, 1977; Buchmann *et al.*, 2010; Gebauer *et al.*, 2002). The leaves of *Adansonia digitata* contain mucilage and provides thickening to soups relished in most part of West Africa e.g. Kuka (northern Nigeria) and Lalo (Mali) and a weaning food in some other places. The mucilage has been classified as a galacturonorhamnan polysaccharide which is acidic due to its high content of uronic acid and also

METHODOLOGY

Adansonia digitata leaves from Baobab trees at Danjibga area of Zamfara State, Nigeria, Ethanol 97% denatured with 3% IPA, (Chem.-Lab NV, Belgium), and Methanol dry, Hydranal[®], (Sigma Aldrich, UK). All other materials were of analytical grade.

Collection, extraction and purification

Leaves of *Adansonia digitata* plucked off from Baobab tree were air dried and processed as described by Mahmud *et al.* (2019). "The crude dried leaves of *Adansonia digitata* were air dried and size reduced to fine powder using a homogenizer then sieved through a 250 µm mesh. A two hundred (200.0) gram weight of the leaf powder was macerated in 4 L of deionised

water, preserved with 0.1 % potassium sorbate and allowed to disperse for a period of 24 h at room temperature of 20 °C. The thickened mucilage was then diluted to 10 L with deionised water and allowed to separate by sedimentation at 6 °C in the refrigerator for 5 days. Thereafter, the mucilaginous supernatant was collected and centrifuged (Multifuge 3S-R, Heraeus, Germany) at 4000 x g for 20 min to separate the fine leaf particulates from the mucilage. The collected mucilage was precipitated with an equal volume of ethanol (97%) and again thoroughly washed with ethanol to remove chlorophyll until it formed a clear rubbery clumped material. The clump was shredded and spread on adsorbent paper to air dry

before packing into beakers and sealed with perforated aluminum foils then dried in a vacuum oven (WTC Binder, Tuttingen, Germany) at 40° C for 48 hours". The resulting cream coloured mucilage was referred to as ADM. To validate this extraction process, ADM was extracted by boiling in distilled water for 1 h as described by Deshmukh *et al.* (2013), to produce a light green colored mucilage (ADM-g).

Physicochemical characterization

Organoleptic properties including color and texture were physically assessed using the sense organs.

pH measurement

The pH_(20°C) of a 1.0 % w/v concentration of ADM prepared in deionized water was determined using a pH meter and the values recorded,.

Aqueous solubility test

This determination was carried out using gravimetric analysis. One (1.0) gram quantity of ADM sample was weighed into 1.0 L of deionised water and allowed to hydrate at room temperature for 24 h. Thereafter, the dispersion was filtered through a pre weighed filter paper (Whatman size 1) of medium porosity. The residue on the filter paper was dried in an oven at 40 °C for 24 h and the differences in weight determined as described by Nep & Conway (2010a).

Moisture content determination

The moisture content of ADM was determined using a DL35 Karl Fischer titrator (Metler Toledo, Beersel, Belgium) and a Metler balance. In this method methanol dry (20 ml) was used as the medium while Hydranal was used as the titrant. A 50 mg weight of ADM was transferred into the system and the percentage water content was determined in triplicate.

Carbon, Hydrogen and Nitrogen (CHN) content

Carbon, Hydrogen and Nitrogen (CHN) method attached to Eager 300 software was carried out in order to determine the elemental composition of the mucilage. A 2.655g quantity of ADM was combusted at 900 °C in presence of excess oxygen. The combustion products were separated by means of programmed temperature desorption system which measured thermal conductivity variations using K factor calibrations.

Determination of apparent viscosities

The viscosities of aqueous dispersions of ADM were measured using a rotational viscometer (Modular Compact Rheometer series 102, PP50, Anton Paar GmbH, Graz, Austria) with a plate-plate technique applying a gap of 1 mm and performed at a constant temperature of 20° C (Willecke *et al.*, 2017).

Concentrations of 1, 2, 3 and 4 % w/v were investigated by applying varying ranges of shear rate from 0.1s⁻¹ to 2000 s⁻¹. Prior to this determination, the dispersions were hydrated for 8 h and agitated on a magnetic stirrer. The viscosities of the various concentrations were recorded.

Thermal analysis

The method of (Monteyne et al., 2016) was adopted with some modifications. A 1.40 g quantity of ADM was carefully weighed and encapsulated in hermetic pans covered with hermetic lids. The degradation, glass transition (T_g) temperatures and melting points (T_m) were measured using Differential scanning calorimeter (DSC Q2000 V24.10, Build 122, Newcastle, USA) connected to a Universal V4.5A TA instrument software. The DSC pan and lid were sealed with special punches and heating was performed at a rate of 10° C/ min while ramping was performed from -20 °C to 250 °C using nitrogen as purge gas.

Attenuated Total Reflectance Fourier Transforms Infrared (ATR -FTIR) Studies

An ATR-FTIR spectroscopy was conducted on the purified ADM powder. Spectra were recorded using an ATR FT-IR spectrometer (Thermo Fisher Scientific, Nicolet iS5 ATR FT-IR spectrometer, Erembodegem-Aalst, Belgium) while a diamond ATR crystal was pressed against the samples. Each spectrum was collected twice in the 4000 - 550 cm⁻¹ range with a resolution of 4 cm⁻¹ and averaged over 128 scans (Claeys *et al.*, 2014). The functional groups present were identified and an identity to the polysaccharide depending on its chemical structure and chain conformation was obtained.

Carbon-13 High-Resolution Solid-State Nuclear Magnetic Resonance (NMR) studies

Carbon-13 solid-state CP/MAS NMR spectra were acquired at ambient temperature on an Agilent VNMRS Direct Drive 400MHz spectrometer (9.4 T wide bore magnet) equipped with a T3HX 3.2 mm probe dedicated for small sample volumes and high decoupling powers. Magic angle spinning (MAS) was performed at 12 kHz with ceramic zirconia rotors of 3.2 mm in diameter (22 µl rotors). The aromatic signal of hexamethyl benzene was used to determine the Hartmann-Hahn condition ($\omega_{1H} = \gamma_H B_{1H} = \gamma_C B_{1C} = \omega_{1C}$) for cross-polarization (CP) and to calibrate the carbon chemical shift scale (132.1 ppm). Acquisition parameters used include a spectral width of 50 kHz, a 90° pulse length of 2.5 µs, a spin-lock field for CP of 100 kHz, a contact time for CP of 1.5 ms, an acquisition time of 20 ms, a recycle delay time of 10 s and 25000 accumulations. High power proton dipolar

decoupling during the acquisition time was set to 100 kHz.

Proton liquid-state NMR

Proton Nuclear Magnetic Resonance (^1H NMR) spectra of solutions in D_2O were recorded at room temperature on a Varian Inova 400 spectrometer using a 5 mm four-nucleus PFG probe. The chemical shift scale (δ) in ppm was calibrated relative to the signal of remaining HOD (4.72 ppm). Free induction decays were collected with water suppression and using the following acquisition parameters: a 90° pulse of 6.35 μs , a spectral width of 6.5 kHz, an acquisition time of 3 s, a preparation delay of 12 s and 128 accumulations. A line-broadening factor of 1 Hz was applied before Fourier transformation to the frequency domain.

Dynamic vapour sorption

The water sorption behavior of ADM powder was measured using the Dynamic Vapor Sorption Equipment (Surface Measurements Systems, Middlesex, UK). During measurement the humidity was increased in 10 % RH steps to 100 % RH for the sorption phase and then decreased in a similar fashion for desorption phase at a temperature of 21°C .

RESULTS AND DISCUSSION

Extraction of ADM from the leaves of Baobab have traditionally been by heat application via dispersion in hot water for 3 min or 2 h at 100°C as given by literature (Woolfe *et al.*, 1977; Deshmukh *et al.*, 2013; Manyarara *et al.*, 2013). Because heat is known to affect the mucilage viscosity (Woolfe *et al.*, 1977), a heat free method was adopted herein. The dried mucilage was a cream colored powder with a percentage yield of $3.5 \pm 0.53\%$, higher than obtained by other researchers (Woolfe *et al.*, 1977; Deshmukh *et al.*, 2013). The extraction and purification with ethanol dissolved the chlorophyll (Sepúlveda *et al.*, 2007). When heat was applied, a light green coloured

Particle shape and size analysis

The analysis of particle size and shape was performed via high order image analysis (QICPIC, Sympatec, Clausthal-Zellerfeld, Germany with GRADIS1.00 63.0 mm - M7). A 5 g quantity of ADM was mounted on a vibratory feeder allowing the particles to fall through the fall shaft. The particles illuminated by laser light were viewed on a screen as they fell on to the extractor surface from where they were collected. The software Windox 5 was used to calculate the particle aspect ratio based on volume, which is defined as the ratio of the minimal to the maximal Feret diameter.

Scanning Electron Microscopy (SEM)

SEM images were recorded with a Scanning electron microscope (Quanta 200F (FEI, Eindhoven, The Netherlands) operated at an acceleration voltage of 20 kV. The powder was deposited onto a carbon carrier substrate. Images of ADM were viewed at different magnifications.

ADM (ADM-g) was obtained with a higher percentage yield of 6.28 % w/w and similar to that obtained by another researcher (Manyarara *et al.*, 2013). The light green color obtained suggests remains of chlorophyll that was not totally removed from the extract and very fine leaf particulates that could not be removed via filtration using a muslin cloth and as a result contributed to the weight of the mucilage. The purity of ADM (heat free method) was ascertained via thermal analysis. ADM-g yielded a broader endothermic peak at 135.46°C characteristic of melting and subsequently degraded at 200°C (Figure 1).

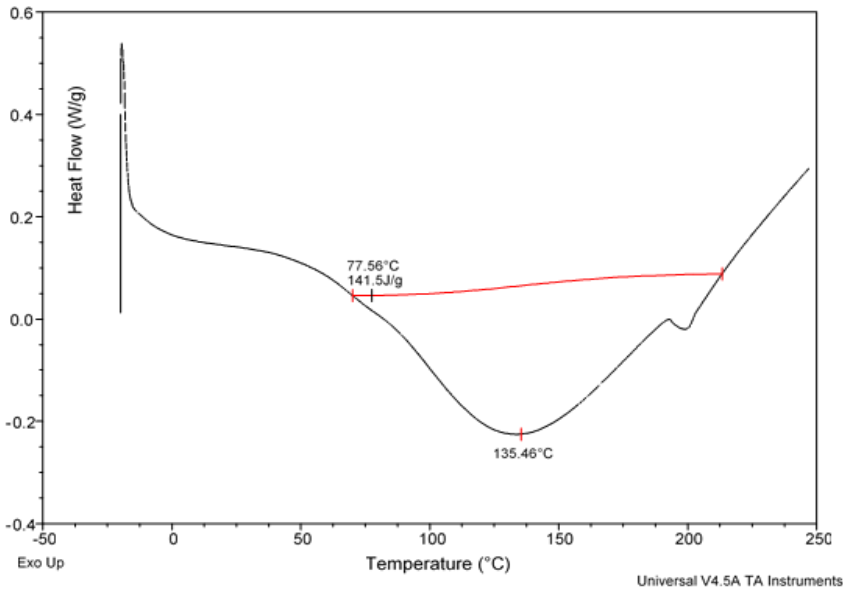


Figure 1. DSC thermogram of Adansonia digitata mucilage (ADM-g) heat treated

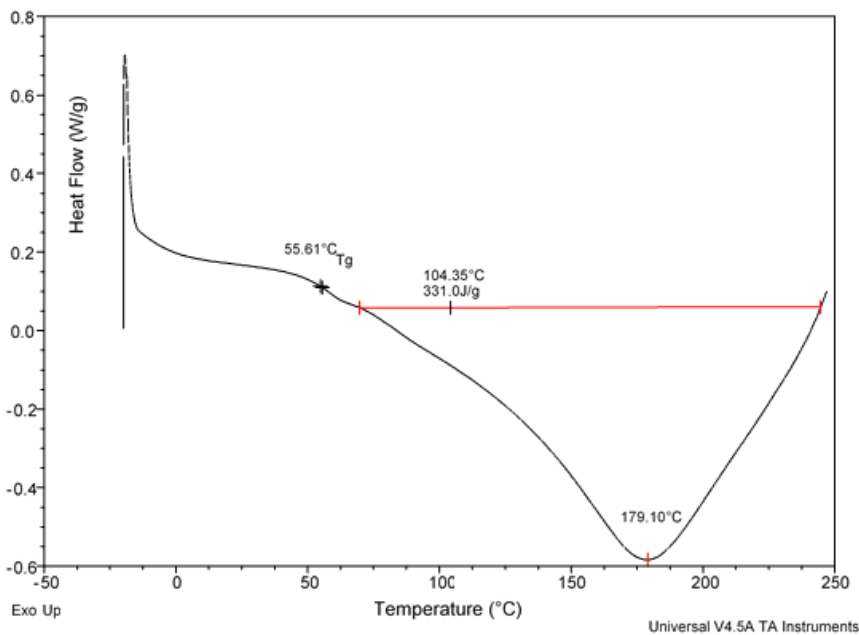


Figure 2. DSC thermogram of Adansonia digitata mucilage (ADM)

The heat free mucilage (ADM) showed a glass transition temperature (T_g) at 55.61 °C and broad endothermic melting curve at 179.10 °C (Figure 2). The low melting temperature of ADM-g indicates presence of impurities. Besides, the purer the substances and the smaller the sample size, the sharper its melting temperature (Widmann *et al.*, 2000; Haji *et al.*, 2006). Impure substances often show several peaks, this was not seen in the ADM thermogram and hence suggested that a level of purification was achieved in the extraction stage when compared with ADM-g. The observed nature of the curve (broad,

concave and T_g of 55.61°C) of ADM is due to its amorphous and partial crystalline nature. Broad curves in DSC thermogram of other polymeric materials have also been reported (Meka *et al.*, 2012). The T_g above room temperature is an indication that powdered ADM will remain stable in the amorphous state at room temperature, i.e. in its glassy state without conversion into crystalline phase due to lack of mobility of the molecules (Buckton, 2021) T_g is a valued characterization parameter of a material which can provide important information regarding the end use performance and properties of that material or product

(Sichina, 2000). When compared to other mucilages, the T_g and T_m of ADM were close to those of Okra mucilage (T_g of 60°C and T_m of 180°C) obtained by (Zaharuddin *et al.*, 2014). It should be noted that this was the first time DSC scans of ADM was performed. The color and purity of ADM is affected by extraction method, hence the heat free/cold extraction method is thereby proposed for ADM extraction. Further discussions are on the heat free ADM only and is referred to as ADM in the text.

The aqueous solubility of ADM could not be determined by the method of Nep & Conway (2010a) as described in the text. This was because on dispersion of ADM in distilled water, already the 1 % mixture hydrated and formed a very viscous gel that was unable to pass through the porous filter paper used. This viscous gelling nature of ADM is similar to findings of Woolfe *et al.* (1977). For the different concentrations to be measured, hydration and stirring was done for over 8 h. The ADM mucilage displayed pseudo plastic characteristics, increasing in viscosity with increasing shear rate (Figure 3) and like many

other polysaccharides, the viscosity increased with increasing mucilage concentration. Mahmud *et al.* (2019) reported that the concentration of ADM polymer in tablet compacts increased viscosity as well as gel layer strength that enabled slow diffusion of drug from the tablet matrix to achieve prolonged release. Furthermore, this viscous nature of ADM prevented a Gel Permeation Chromatography (GPC) to be recorded when distilled water was used as the solvent requiring a solvent to be solubilized in. (Jani *et al.*, 2009) mentioned that mucilages are composed of several sugar monomers and do not dissolve but form slimy mass, viscous solutions or gels in water. This is quite true for ADM regarding the sugars detected via NMR. Furthermore, the nature of the compounds present in the mucilage influence these properties. This viscous nature of ADM has enabled its use as a suspending agent, thickener, binder in tablet formulations and matrices for modified release dosage forms. In addition, ADM can be useful as an emulgent as well as in topical applications such as gels.

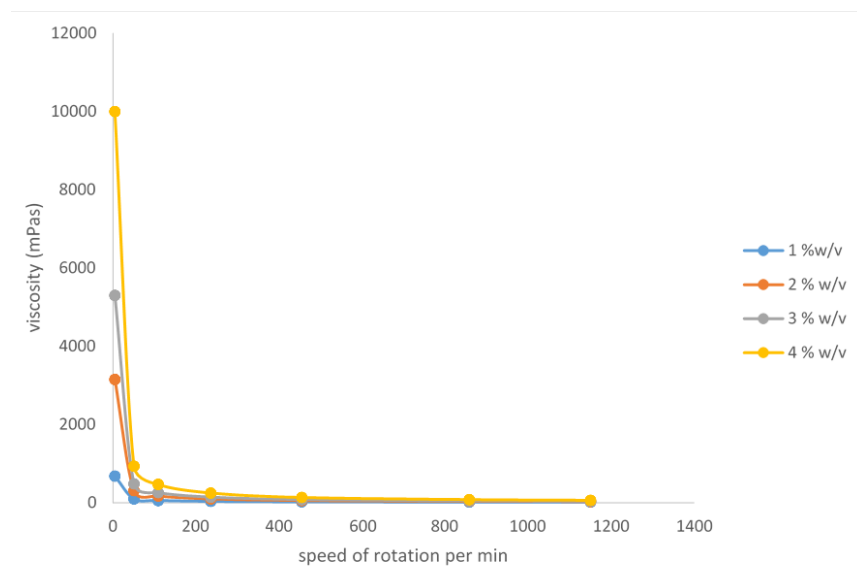


Figure 3. Viscosity of various concentrations of ADM dispersion

T

he acidic nature of *Adansonia digitata* mucilage (Table I) has also been reported by (Woolfe *et al.*, 1977). This supports the assertion by (van Aken, 2006) that plant mucilages are largely acidic polysaccharides. The acidic nature of ADM is an indication that it can provide protection for drugs and remains stable in the very acidic environment of the stomach, as a result of its acidic groups that will not ionize at this pH. The high moisture content indicated that ADM is hygroscopic. The high percentage of moisture could be due to adsorption of water

molecules that may form single or more layers on the surface of the solid or due to entrapped liquid within the capillaries of the solid as a result of which it cannot evaporate easily (Aulton, 2007). This was confirmed by water sorption studies which described the mechanism of water uptake by ADM to be, an initial surface penetration (adsorption) at a low relative humidity (RH 10%) above which bulk penetration (absorption) dominated. As a result, slow diffusion of water from the sample during desorption was observed (Figure 4). This resulted in entrapment of water

molecules into ADM structure due to re-crystallization caused by moisture induced lowering of glass transition (T_g) of amorphous regions in the sample as observed during desorption and defined by rapid increase in molecular mobility of ADM at 70 % RH and a rapid desorption in a similar manner . A marked hysteresis between RH 10 % and RH 80 % in the corresponding water sorption isotherm (Figure 5) further explained the extent of water absorption. This gap that remains over the entire partial pressures range is characteristic of pharmaceutical and food hysteresis shape. Kapsalis (1987) mentioned that partially amorphous materials exhibit a considerable bulk

absorption of water which in some cases is accompanied by swelling effects. This was observed in the moisture sorption isotherm of ADM and swellings from 0.05% at 344 min up to 59.86% after 2488 min. ADM swelling was also described elsewhere to be the reason for ADM prolonged drug release for over 12 h (Mahmud *et al.*, 2019). Again, the water sorption studies confirmed the partial amorphous nature of ADM previously seen in DSC thermogram as broad curves in addition to amorphous behavior of exhibiting a T_g . Burnett *et al.* (2006), have mentioned correlations between DVS and DSC regarding T_g of some materials.

Table I: Physicochemical properties of purified *Adansonia digitata* mucilage (ADM)

Parameters	Results
Colour of crude	Green
Colour of purified	Cream
pH (21°C)	6.07
Texture	Fine
Percentage yield (% w/w)	3.5 ± 0.53
Viscosity (20°C) (mpas of 2% w/v)	3,160
(mpas of 4% w/v)	10,000
Aqueous solubility	ND
Moisture content (%)	12.91
Particle size (µm) x10	208.14
x50	663.57
x90	1372.00
Aspect ratio (a10)	0.48
(a50)	0.66
(a90)	0.82
Sphericity (s10)	0.70
(s50)	0.80
(s90)	0.87
Shape (SEM)	Needle like - irregular elongated fibres
Proximate values (%)	
Nitrogen	0.64
Carbon	34.95
Hydrogen	5.55

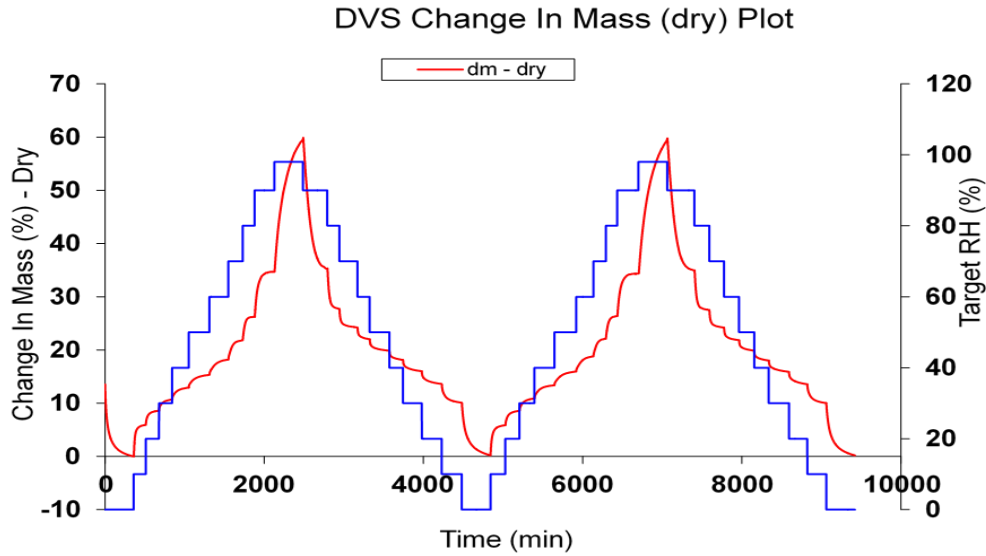


Figure 4. Water sorption kinetics for *Adansonia digitata* mucilage (ADM) at 21°C

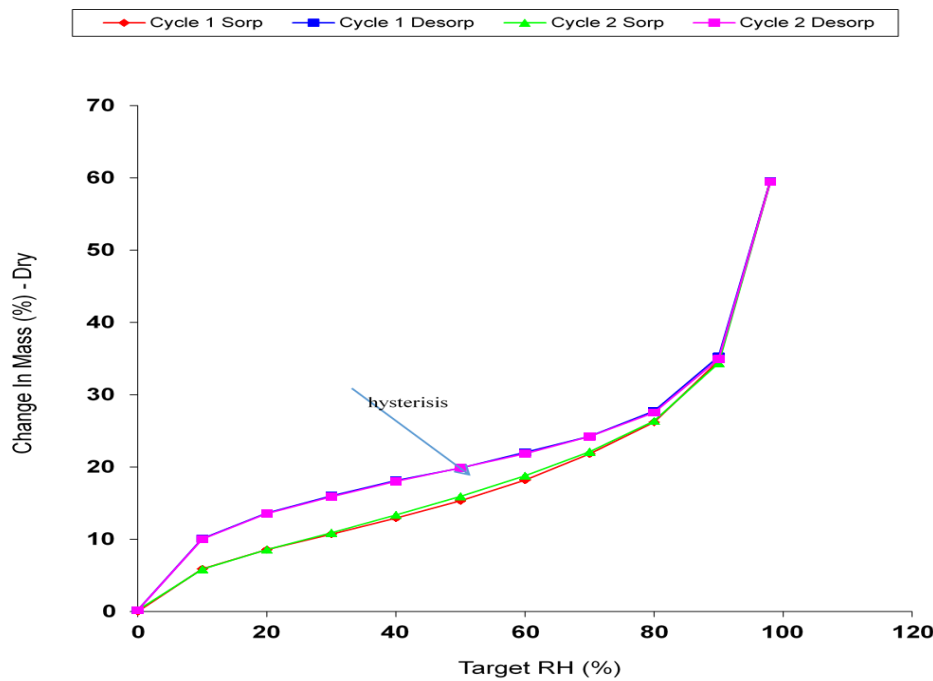


Figure 5. Moisture sorption isotherm of *Adansonia digitata* mucilage (ADM) at 21°C

High water content promotes biocompatibility of hydrogel due to similarities to the natural composition and mechanical strength of its extracellular matrix, especially those that are carbohydrate-based (Hoare & Kohane, 2008). The Baobab plant, *Adansonia digitata* has been found to be among the most effective plants that prevent water loss (Gebauer *et al.*, 2002). That said, ADM requires to be processed in low humidity conditions. Additional packaging material or conditions such as “tightly sealed in a cool, dry place”

should be used to prevent it from degradation on storage and also to improve its economic importance and industrial application.

The only technique that measures multiple values of particles is microscopy or automated image analysis of which QICPIC is an example. The procedure provided valid statistical and quality information on number and shape of particles imaged. Several modes appeared due to presence of large aggregates or long thin fibrous particles in the frequency distribution curve of ADM

(Figure 6), which relates to characteristic crumbled nature of the mucilage when precipitated and dried. The particle size distribution (PSD) was described by two points (x10 and x90) in addition to one of the central values (x50 median) as shown in Table I. ADM constituted 10 % small particles and 90 % large particulates. Aspect ratio of 0.82 indicated that 90 % of small particles had the highest aspect ratio which decreased as the particle sizes increased, due to increase in perimeter of the irregular shaped particles. A 10 % of the sample deviated from the ideal length: diameter with an aspect ratio of 0.48. An ideal case will assume a shape factor of 1 while values close to 0 represent great deformation such as curled fibers (Wojnar *et al.*, 2000.). Median sphericity (another shape factor) further described 90 % of the particles

tended towards unity while 10 % remained irregular. This showed a mixture of spherical and curled fibers (Figure 7) and may be less prone to segregation when considered in tablet manufacture. The external features of ADM particles determined by QICPIC corroborated well with the SEM images (Figure 7) that revealed elongated irregular and curled fibrous particles which when related to Qicpic image analysis attributed to low aspect ratio value of 0.48 and median particle diameter of 663.57 μm . Elemental analysis revealed Carbon, Hydrogen and Nitrogen (Table I). The nitrogen content obtained indicated the presence of amino acids which have been reported to be present in the leaves of *Adansonia digitata* (Ogbaga *et al.*, 2017).

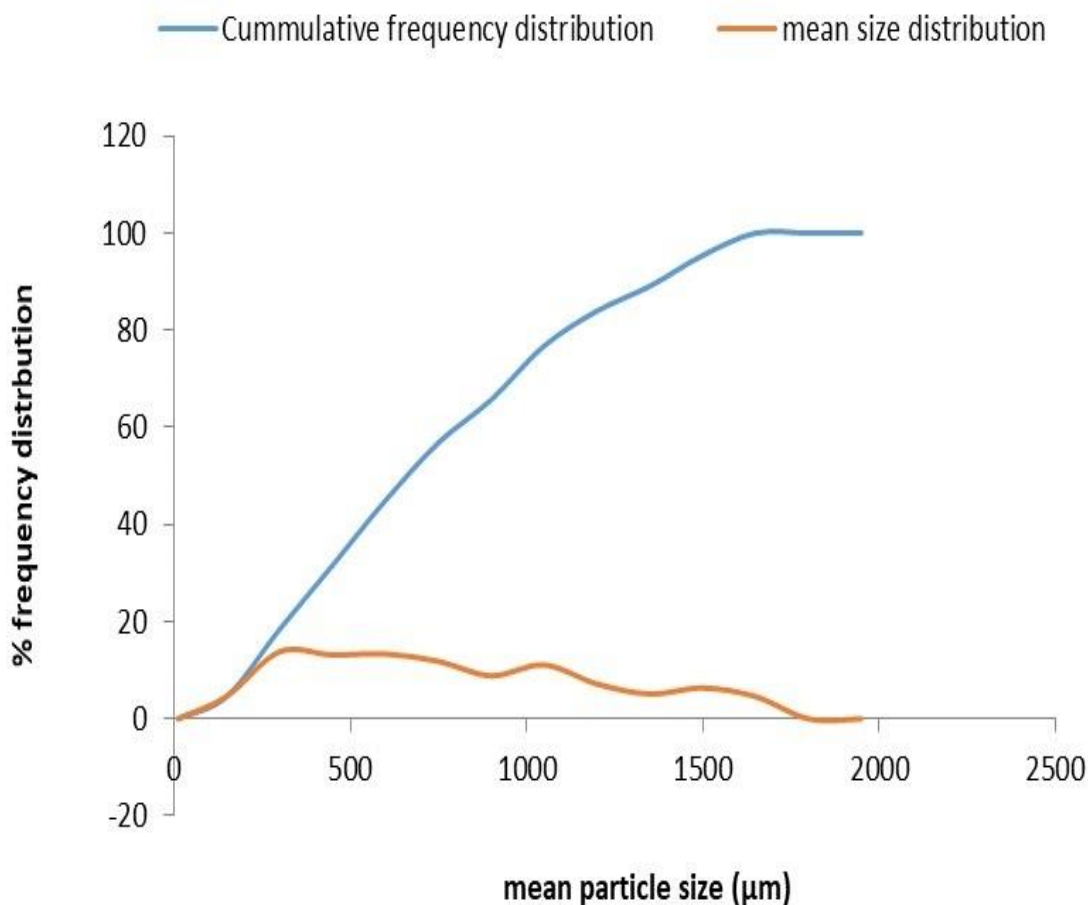


Figure 6. Cumulative percent frequency distribution curves (undersize) of unmilled ADM powder via QICPIC

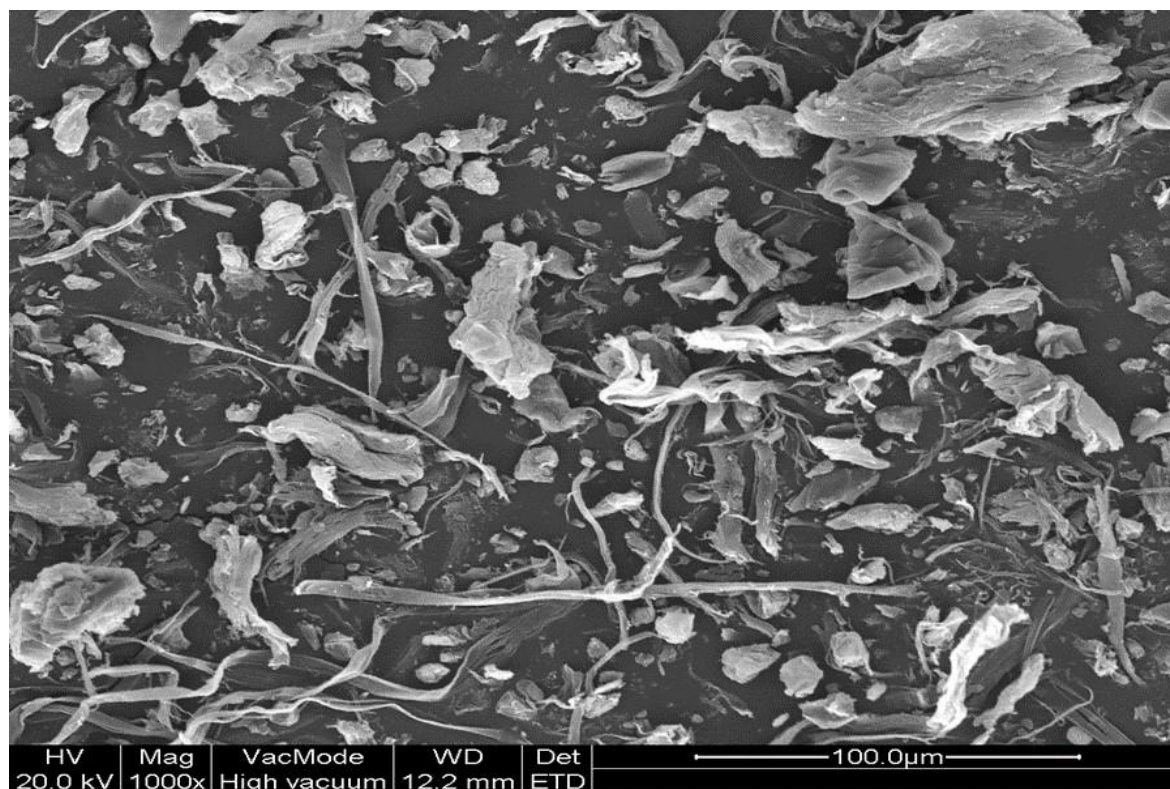


Figure 7. SEM image of *Adansonia digitata* mucilage (ADM) at x1000 magnification

ATR-FTIR offers the advantage of analyzing samples directly without dilution in a few seconds. Carbonyl, methyl, amino and carboxylic groups were the major functional groups identified in the spectra of ADM at different wavelength penetrations. Absorption peaks at 633 cm^{-1} depicted the presence of N-H primary amides, at $1100 - 950\text{ cm}^{-1}$ depicted possible presence of thioamides N-C=S and $\text{CH}_3\text{-C}$ aliphatic and saturated aliphatic ethers. Band stretching at 1239 cm^{-1} suggested C-O carboxylic acid dimers, and esters CH_3COOR . In relation to sugars it represents aromatic compounds of rhamnose and galactose. Band at 1416 cm^{-1} showed the possibility of presence of N=N aromatic azo compound and C-O stretch while band at 1600 cm^{-1} depicted presence of N-H: primary amide and band at 1725 cm^{-1} depicted C=O: stretch of different types (saturated aliphatic ketones, saturated aliphatic aldehydes, aryl esters, formates and saturated aliphatic carboxylic acid dimers)

characteristic of polysaccharides which may relate to galacturonic acid. (Abdulsamad *et al.*, 2015) and (Saeedi *et al.*, 2013) have reported C=O at this regions for cashew gum and plantago mucilage. ATR-FTIR result as well as elemental analysis indicated the presence of amide groups and nitrogen in ADM. Moreover, azo group containing polymers can be considered for enzyme triggered release in colon specific drug delivery (Jain *et al.*, 2007; Perrie & Rades, 2012).

^{13}C NMR spectrum of mucilage confirmed the presence of these sugars. It showed chemical shifts characteristic of carbohydrates at 62.49, 71.35, 81.67, and 104.30. The signals at 71.35 ppm can be attributed to CH group of arabinose. ^{13}C NMR spectrum shows that for C-6, the mucilage exhibited the characteristic signal at d 173.98 of carboxyl in acid form of galacturonic acid.

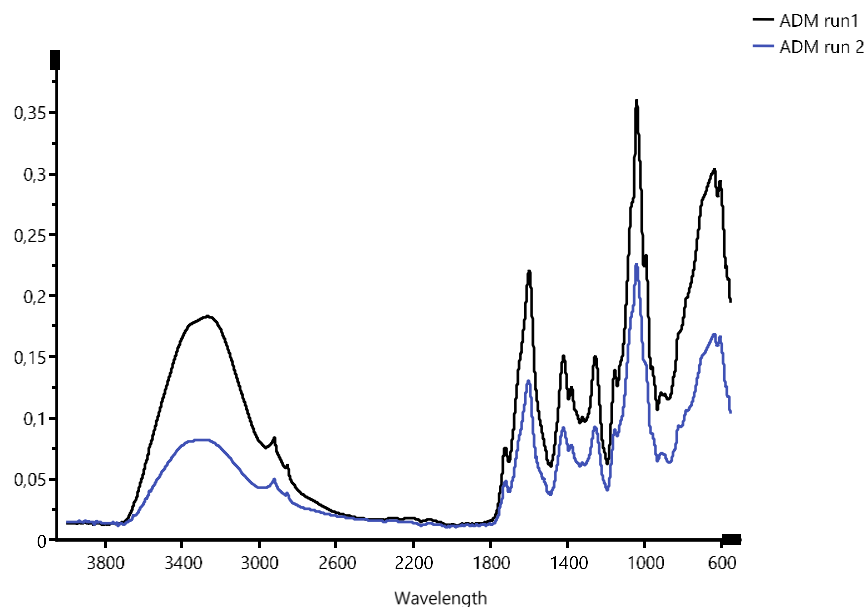


Figure 8. Attenuated Total Reflectance Fourier Infra-Red (ATR-FTIR) spectrum of ADM Solid

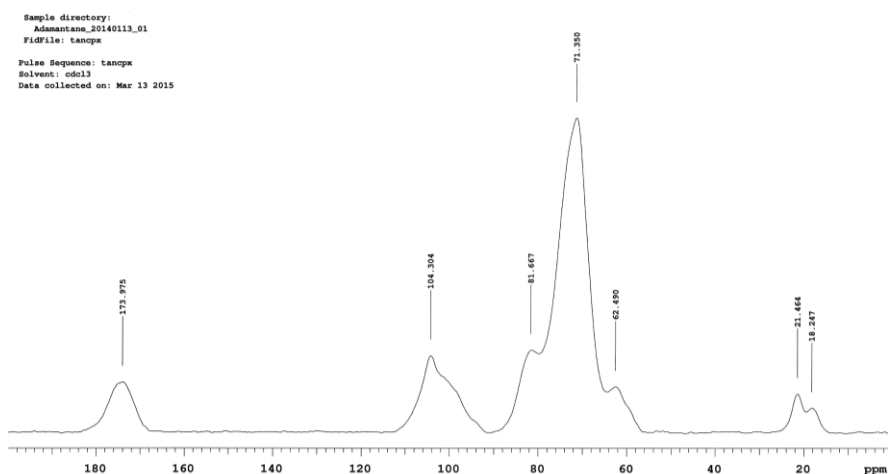


Figure 9. Solid state ¹³C Nuclear magnetic resonance spectrum of ADM

The signals at 18.25 and 21.46 may be due to methyl group of rhamnose (Petera *et al.*, 2015). The ¹H NMR spectrum (not shown) for mucilage showed few singlet's at high field (δ 1.22 ppm (s), δ 1.98 ppm (s), which indicates methyl groups of rhamnose. Signals at 3.6, 3.7, 3.9 and 4.2 suggests galactose derivatives. Protons were assigned to β -sugar residues (d 4.7-4.8 ppm) and the α -sugar residue (δ 5.0-5.2 ppm). The two signals observed (δ 4.0 and δ 3.8 ppm) in the ¹H NMR spectrum of mucilage assigned to glucose group (Sims *et al.*, 2018). The ¹H NMR spectrum showed peaks between 3.4 to 5.18ppm are characteristic of sugar residues (Ma & Pawlik, 2007). The ¹H NMR spectrum for mucilage showed signals at 5.2 (d), 5.18 (d), 4.5

(t), and various signals at 3.50 -4.1) which suggests the presence of arabinose (Ibraheem *et al.*, 2021). Keys; s=singlet, d=doublet, t=triplet, m multiplet.

Although research works on *Adansonia digitata* mucilage are few, the viscosity of ADM found in this research agreed with findings of previous works (Woolfe *et al.*, 1977). However, the pseudo plastic behavior and apparent viscosity of the mucilage as it varied with increasing concentration for non-Newtonian fluids is described only herein. The sugar compounds of ADM have been previously reported except for arabinose reported herein. The SEM, DSC scan, particle sizes via QICPIC and water sorption studies via DVS describing the mechanism of sorption are a first-time report on ADM.

CONCLUSION

A proposed heat free extraction method has been established. A well-established correlation was obtained between the extraction technique and the physicochemical characteristics of *Adansonia digitata* mucilage (ADM). The mechanism of water sorption of the partially amorphous mucilage was found to be by both surface sorption and bulk absorption. The unmilled ADM was characterized by needle like and

irregular elongated fibres. Although a low yield of ADM mucilage was obtained, its highly viscous, fast hydration and gelling properties, glass transition above room temperature indicated that it has potential for many pharmaceutical applications including binding, coating, gelling agents as well as matrix forming potentials.

ACKNOWLEDGEMENTS

We appreciate Prof. JeanPaul Remon and Dr. Jurgen Vercruyse of the Laboratory of Pharmaceutical Technology Ghent University, Belgium for their support in the conduct of this research. We appreciate Pharm. Aliyu Hamidu Ahmed of Department of Pharmacognosy, Usmanu Danfodio University

Sokoto, Nigeria for the interpretation of NMR spectra. Tertiary Education Trust Fund (TETFUND) Nigeria for the award of research grant and Ahmadu Bello University for facilitating the award are much recognized.

REFERENCES

- Abdulsamad, A., Oyi, A. R., Isah, A. B., & Odidi, I. (2015). Derivatisation of Cashew Gum by Esterification Using Citric Acid and Glycerol. *British Journal of Pharmaceutical Research*, 6(3).
- Aulton, M. E. (2007). Aulton's pharmaceuticals: The design and manufacture of medicines. 3rd. *Chapter*, 21, 298–299.
- Buchmann, C., Prehsler, S., Hartl, A., & Vogl, C. R. (2010). The importance of baobab (*Adansonia digitata* L.) in rural West African subsistence—Suggestion of a cautionary approach to international market export of baobab fruits. *Ecology of Food and Nutrition*, 49(3), 145–172.
- Buckton, G. (2021). Solid-state properties. *Aulton's Pharmaceuticals E-Book: The Design and Manufacture of Medicines*, 114.
- Builders, P. F., Okeke, U., & Egieye, A. S. (2007). Evaluation of the controlled release potential of. *Journal of Phytomedicine and Therapeutics*, 12, 22–30.
- Burkill, H. M. (1995). The useful plants of west tropical Africa, Vols. 1-3. *The Useful Plants of West Tropical Africa, Vols. 1-3.*, 2. ed.
- Chadare, F. J., Linnemann, A. R., Hounhouigan, J. D., Nout, M. J. R., & Van Boekel, M. (2008). Baobab food products: A review on their composition and nutritional value. *Critical Reviews in Food Science and Nutrition*, 49(3), 254–274. <https://doi.org/10.1080/10408390701856330>
- Claeys, B., De Bruyn, S., Hansen, L., De Beer, T., Remon, J. P., & Vervaet, C. (2014). Release characteristics of polyurethane tablets containing dicarboxylic acids as release modifiers—a case study with diprophylline. *International Journal of Pharmaceutics*, 477(1–2), 244–250.
- Deshmukh, S. S., Katare, Y. S., Shyale, S. S., Bhujbal, S. S., Kadam, S. D., Landge, D. A., Shah, D. V., & Pawar, J. B. (2013). Isolation and Evaluation of Mucilage of *Adansonia digitata* Linn as a Suspending Agent. *Journal of Pharmaceutics*, 2013. <http://dx.doi.org/10.1155/2013/379750>
- Gebauer, J., El-Siddig, K., & Ebert, G. (2002). Baobab (*Adansonia digitata* L.): A review on a multipurpose tree with promising future in the Sudan. *Gartenbauwissenschaft*, 67(4), 155–160.
- Haji, M. D. N. A. B., Bin, M. P. M. M. K., & Aziz, A. (2006). *HIGH SENSITIVITY DIFFERENTIAL SCANNING CALORIMETER (HSDSC) TECHNIQUE FOR ASSAYING GINGER OLEORESIN*. Universiti Teknologi Malaysia, Kuala Lumpur.
- Hoare, T. R., & Kohane, D. S. (2008). Hydrogels in drug delivery: Progress and challenges. *Polymer*, 49(8), 1993–2007.
- Ibraheem, S., Idris, Y. M. A., Mustafa, S., Kabeir, B., Abas, F., Maulidiani, M., & Abdul Hamid, N. (2021). Phytochemical profile and biological activities of Sudanese baobab (*Adansonia digitata* L.) fruit pulp extract. *International Food Research Journal*, 28(1).
- Jain, A., Gupta, Y., & Jain, S. K. (2007). Perspectives of biodegradable natural polysaccharides for site-specific drug delivery to the colon. *J Pharm Pharm Sci*, 10(1), 86–128.
- Jani, G. K., Shah, D. P., Prajapati, V. D., & Jain, V. C. (2009). Gums and mucilages: Versatile excipients for pharmaceutical formulations. *Asian J Pharm Sci*, 4(5), 309–323.
- Kapsalis, J. G. (1987). *Objective methods in food quality assessment*. CRC Press.
- Ma, X., & Pawlik, M. (2007). Intrinsic viscosities and Huggins constants of guar gum in alkali metal chloride solutions. *Carbohydrate Polymers*, 70(1), 15–24.

- Mahmud, H. S., Vercruyssen, J., & Remon, J. (2019). In vitro and in vivo assessment of Adansonia digitata mucilage as a matrix former in modified release tablets of metoprolol tartrate. *Journal of Drug Delivery Science and Technology*, 54, 101227. <https://doi.org/10.1016/j.jddst.2019.101227>
- Manyarara, T., Chifamba, J., & Derera, P. (2013). Investigation of the suspending properties of Dicerocaryum zanguebarium and Adansonia digitata mucilage as structured vehicles.
- Meka, V. S., Nali, S. R., Songa, A. S., & Kolapalli, V. R. M. (2012). Characterization and in vitro drug release studies of a natural polysaccharide Terminalia catappa gum (Badam gum). *AAPS PharmSciTech*, 13(4), 1451–1464.
- Monteyne, T., Adriaensens, P., Brouckaert, D., Remon, J.-P., Vervaet, C., & De Beer, T. (2016). Stearic acid and high molecular weight PEO as matrix for the highly water soluble metoprolol tartrate in continuous twin-screw melt granulation. *International Journal of Pharmaceutics*, 512(1), 158–167.
- Nep, E. I., & Conway, B. R. (2010a). Characterization of grewia gum, a potential pharmaceutical excipient. *Journal of Excipients and Food Chemicals*, 1(1), 30–40.
- Nep, E. I., & Conway, B. R. (2010b). Polysaccharide gum matrix tablets for oral controlled delivery of cimetidine. *Journal of Pharmaceutical Sciences and Research*, 2(11), 708–716.
- Ogbaga, C. C., Nuruddeen, F. A., Alonge, O. O., & Nwagbara, O. F. (2017). Phytochemical, elemental and proximate analyses of stored, sun-dried and shade-dried baobab (Adansonia digitata) leaves. *2017 13th International Conference on Electronics, Computer and Computation (ICECCO)*, 1–5.
- Perrie, Y., & Rades, T. (2012). *FASTTrack Pharmaceutics: Drug Delivery and Targeting*. Pharmaceutical press.
- Petera, B., Delattre, C., Pierre, G., Wadouachi, A., Elboutachfai, R., Engel, E., Poughon, L., Michaud, P., & Fenoradosoa, T. A. (2015). Characterization of arabinogalactan-rich mucilage from Cereus triangularis cladodes. *Carbohydrate Polymers*, 127, 372–380.
- Saeedi, M., Morteza-Semnani, K., & Sagheb-Doust, M. (2013). Evaluation of Plantago major L. seed mucilage as a rate controlling matrix for sustained release of propranolol hydrochloride. *Acta Pharmaceutica*, 63(1), 99–114.
- Sepúlveda, E., Sáenz, C., Aliaga, E., & Aceituno, C. (2007). Extraction and characterization of mucilage in Opuntia spp. *Journal of Arid Environments*, 68(4), 534–545. <https://doi.org/10.1016/j.jaridenv.2006.08.001>
- Sibibe, M., & Williams, J. T. (2002). Baobab–Adansonia digitata Fruits for the future. *Int Centre Underutil Crops. University of Southampton, UK*.
- Sichina, W. J. (2000). Measurement of T_g by DSC. *PerkinElmer, Application Note*.
- Sims, I. M., Smith, A. M., Morris, G. A., Ghorri, M. U., & Carnachan, S. M. (2018). Structural and rheological studies of a polysaccharide mucilage from lacebark leaves (Hoheria populnea A. Cunn.). *International Journal of Biological Macromolecules*, 111, 839–847.
- van Aken, G. A. (2006). 15 Polysaccharides in Food Emulsions. *Food Polysaccharides and Their Applications*, 521.
- Wickens, G. E. (1982). The baobab: Africa's upside-down tree. *Kew Bulletin*, 173–209. <https://doi.org/10.2307/4109961>
- Widmann, J., Schubnell, M., Riesen, R., Schawe, J., Darribère, C., & Jörimann, U. (2000). Interpreting DSC curves: Part 2: Isothermal measurements. *UserCom*, 2, 1–10.
- Willecke, N., Szepes, A., Wunderlich, M., Remon, J. P., Vervaet, C., & De Beer, T. (2017). Identifying overarching excipient properties towards an in-depth understanding of process and product performance for continuous twin-screw wet granulation. *International Journal of Pharmaceutics*, 522(1–2), 234–247.
- Wojnar, L., Friel, J., Grande, J., Hetzner, D., Kurzydowski, K., & Laferty, D. (n.d.). Practical Guide to Image Analysis. Ohio, USA: ASM International; c2000. Chapter 7. *Analysis and Interpretation*, 145–182.
- Woolfe, M. L., Chaplin, M. F., & Otchere, G. (1977). Studies on the mucilages extracted from okra fruits (Hibiscus esculentus L.) and baobab leaves (Adansonia digitata L.). *Journal of the Science of Food and Agriculture*, 28(6), 519–529. <https://doi.org/10.1002/jsfa.2740280609>
- Zaharuddin, N. D., Noordin, M. I., & Kadivar, A. (2014). The use of Hibiscus esculentus (Okra) gum in sustaining the release of propranolol hydrochloride in a solid oral dosage form. *BioMed Research International*, 2014.
- Zahra'u, B., Mohammed, A. S., Ghazali, H. M., & Karim, R. (2014). Baobab tree (Adansonia digitata L) parts: Nutrition, applications in food and uses in ethno-medicine—a review. *Ann Nutr Disord & Ther*, 1(3), 1011.

*Address for correspondence: H.S. Mahmud
Department of Pharmaceutics and Industrial
Pharmacy, Ahmadu Bello University, Zaria, Nigeria
Telephone: +234 8035 903 870
E-mails: mhszubair@gmail.com

Conflict of Interest: None declared
Received: February 23, 2023
Accepted: April 14, 2023