

# Effect of ultrasonication on extraction yield, and the rheological and physicochemical characteristics of Mucuna sloanei gum

Article

Published Version

Creative Commons: Attribution 4.0 (CC-BY)

Open Access

Adeboye, A. S., Amiri Rigi, A. ORCID: https://orcid.org/0000-0002-6932-7939 and Emmambux, N. M. (2025) Effect of ultrasonication on extraction yield, and the rheological and physicochemical characteristics of Mucuna sloanei gum. International Journal of Biological Macromolecules, 307 (Part 1). 141794. ISSN 1879-0003 doi: https://doi.org/10.1016/j.ijbiomac.2025.141794 Available at https://centaur.reading.ac.uk/122004/

It is advisable to refer to the publisher's version if you intend to cite from the work. See <u>Guidance on citing</u>.

To link to this article DOI: http://dx.doi.org/10.1016/j.ijbiomac.2025.141794

Publisher: Elsevier

All outputs in CentAUR are protected by Intellectual Property Rights law, including copyright law. Copyright and IPR is retained by the creators or other copyright holders. Terms and conditions for use of this material are defined in the <u>End User Agreement</u>.



# www.reading.ac.uk/centaur

# CentAUR

Central Archive at the University of Reading

Reading's research outputs online

Contents lists available at ScienceDirect



International Journal of Biological Macromolecules

journal homepage: www.elsevier.com/locate/ijbiomac



# Effect of ultrasonication on extraction yield, and the rheological and physicochemical characteristics of *Mucuna sloanei* gum



Adedola S. Adeboye<sup>a</sup>, Atefeh Amiri-Rigi<sup>a,b,\*</sup>, Naushad M. Emmambux<sup>a</sup>

<sup>a</sup> Department of Consumer and Food Sciences, Faculty of Natural and Agricultural Sciences, University of Pretoria, PO Box 0028, Pretoria, South Africa <sup>b</sup> Department of Food and Nutritional Sciences, University of Reading, Whiteknights, Reading RG6 6AP, United Kingdom

#### ARTICLE INFO

Keywords: Extraction yield Mucuna sloanei gum Rheological properties Sugar composition Thermal properties Ultrasound-assisted extraction

# ABSTRACT

Mucuna sloanei flour is widely used as a thickening agent in Southern Nigerian cuisine, contributing to its texture and nutritional value. Additionally, it is gaining attention in pharmaceuticals for its health benefits, biodegradability, biocompatibility, and role in pharmaceutical formulation and controlled-release systems. However, there is a notable gap in the understanding of Mucuna gum extraction and its characterisation. This study investigated ultrasound-assisted extraction to improve gum extraction yield, as well as the physicochemical and rheological properties of M. sloanei gums. The ultrasound process improved extraction yields to 45.81 % and 47.09 % at 30 % and 60 % amplitudes, respectively, compared to 41.5 % for unsonicated extraction, reflecting enhancements of approximately 10.2 % and 13.3 % in yield while preserving or enhancing thermal properties. Thermal analysis indicated gelatinisation temperatures ranging from 92.12 to 103.40 °C, with no significant differences (p > 0.05) among gums. Thermogravimetric analysis revealed that extracted gums exhibited higher degradation onset temperatures than raw flour, indicating enhanced thermal stability which seems promising for industrial application. Furthermore, sonication reduced levels of D-xylose, D-fructose, L-(+)-arabinose, and sorbitol. Viscoelastic testing showed that the extracted gums had reduced shear-thinning behaviour and greater yield stress than the flour, suggesting their effectiveness as binders and viscosifiers. These findings underscore the potential of Mucuna sloanei gum as a commercially viable thickener in food formulations where flour is predominantly used and in the pharmaceutical industry as a natural binder, disintegrant, and controlled-release agent. The study also highlights the efficiency of ultrasound-assisted extraction as a scalable and sustainable method for gum extraction and modification.

# 1. Introduction

Hydrocolloids (HC), widely known as gums, refer to a diverse group of long-chain polysaccharides and some proteins that have the characteristic property of forming gels when dispersed in water. They have a neutral taste/aroma thus they are ideal additives in the food industry for functional roles as substitutes for sugar, fat, or gluten, stabilisers for foam/and or emulsion, modifiers for gelatinisation, inhibitors of crystallisation, and thickening agents. Sources include sap of trees, extract from seeds and seaweeds, microbial gums, and extract from tubers or plant parts [1].

*Mucuna sloanei* (*M. sloanei*) seeds are known to contain a significant amount of hydrocolloids, primarily long-chain polysaccharides, that can form gels when dispersed in water. These hydrocolloids contribute to enhanced functional properties, such as texture, viscosity, and stability in food products, making them valuable for developing innovative food options [2]. *Mucuna sloanei* commonly called "Ukpo" in South-Eastern Nigeria, is extensively used for thickening traditional soups among Ibo communities of the country. The seeds in flour form are added in small quantities to the soup and stirred to solubilise at high temperatures, typically around 70–90 °C. Upon solubilising in the soup, the gum in the flour swells, increasing the thickness of the soup. This traditional application highlights its potential as a natural thickener and texture enhancer in modern food applications, particularly in processed or "convenience foods" [3].

Beyond its culinary uses, studies on *Mucuna* species, including *Mucuna sloanei*, have explored its health benefits and potential for drug delivery systems. In a study by Ugwu et al. (2018), *Mucuna sloanei* seed

https://doi.org/10.1016/j.ijbiomac.2025.141794

Received 19 November 2024; Received in revised form 9 February 2025; Accepted 4 March 2025 Available online 7 March 2025

0141-8130/© 2025 The Authors. Published by Elsevier B.V. This is an open access article under the CC BY license (http://creativecommons.org/licenses/by/4.0/).

<sup>\*</sup> Corresponding author at: Department of Consumer and Food Sciences, Faculty of Natural and Agricultural Sciences, University of Pretoria, PO Box 0028, Pretoria, South Africa.

E-mail address: a.amiririgi@reading.ac.uk (A. Amiri-Rigi).

extract offered hepato-protective benefits by reducing alanine aminotransferase levels and nephroprotective effects through lowering serum urea, blood urea nitrogen, and creatinine levels when consumed in moderate amounts [4]. Modified gelatin capsules containing biopolymer-based microparticles made from Mucuna sloanei gum and oils (liquid paraffin and soybean) demonstrated encapsulation efficiencies of 52.64 % to 82.83 % for aceclofenac. The capsules delayed the drug release in the upper gastrointestinal tract and sustained it in the lower tract, achieving high anti-inflammatory activity (77-86 %). The study demonstrated the potential of Mucuna sloanei gum to enhance colonic drug delivery [5]. Another study on Mucuna gum microspheres demonstrated that crosslinking significantly enhanced drug loading efficiency and sustained release of glibenclamide, highlighting its potential as a reliable oral delivery system [6]. These suggest that Mucuna sloanei's polysaccharides could be applied in pharmaceutical formulations, offering potential health benefits and controlled drug delivery, highlighting its value not only as a food ingredient but also in healthrelated applications.

Despite the health benefits and potential applications of *M. sloanei* in food innovation, pharmaceutical formulations, and drug delivery systems, research attention has been limited, particularly regarding the physicochemical and rheological characterisation of its gum and flour, as well as the extraction process [2,4-8]. Oladipo et al. (2013) [8] successfully extracted, isolated, and purified gum from M. sloanei seeds, which exhibited an emulsion capacity of 41.66 %, freeze-thaw stability of 96.63 %, a bulk density of 0.538 g/ml, and increased viscosity at higher concentrations. Hydrocolloids extracted from M. sloanei in another study by Uzomah and Odusanya (2011) [2] showed high protein (23.92 %) and fat (6.57 %) content, with a wide gelatinisation temperature range (29.52 °C to 98.0 °C). Eke-Ejiofor and Awajiogak (2019) [7] assessed the physicochemical and functional properties M. sloanei (ukpo) flour without extracting the gum, revealing an increase in amylose content with boiling, while exhibiting varying functional properties such as bulk density, solubility, and foam capacity depending on the processing method. In another study, gums extracted from M. sloanei demonstrated potential as stabilisers in food products like beef burgers at concentrations of 0.25 %, 0.5 %, 0.75 %, and 1.0 %, improving water holding capacity, reducing shrinkage, and enhancing texture and sensory properties without compromising product quality [9]. However, the extraction methods and yields were not detailed, and the physicochemical and rheological properties of the gum have not been characterised.

Ultrasonication has emerged as a green and efficient method in speeding up the rate of diffusion and thus, the extraction yield of hydrocolloids [10]. When ultrasonic energy is applied to solutions, it induces a phenomenon known as acoustic cavitation (implosion) [11]. The implosion of bubbles formed in solutions leads to the formation of very high localised temperature and pressure differentials, known as localised "hot spots". These high-energy environments created by ultrasound facilitate the release of analytes into the solution at a shorter time compared to unassisted extraction [12,13]. Therefore, ultrasound assisted extraction using a probe system might serve as an effective method to improve the yield and efficiency of *M. Sloane* gum extraction, with the potential to improve its functional properties for food applications.

However, existing studies lack comprehensive data on the physicochemical composition of the extracted gum and its response to both large and small deformation forces, both of which are crucial for understanding the rheological behaviour of *M. sloanei* gum. Additionally, reported gum yield from defatted cotyledons varies significantly, ranging from 13 % [8] to 33 % [14], which are relatively low and pose sustainability concerns, particularly considering the labour-intensive extraction process. These challenges hinder the scalability and commercialisation of *M. sloanei* gum as a viable food hydrocolloid. Furthermore, ultrasound-assisted extraction (UAE) has shown promise in improving rheological and physicochemical properties, as well as enhancing extraction efficiency by improving diffusion rates and overall yield [10,12]. However, its impact on the functional properties and yield of *M. sloanei* gum remains unexplored. Addressing the gap in knowledge regarding *M. sloanei* as an underutilised African indigenous crop is crucial in regions heavily dependent on imported, unsustainable resources, where developing local alternatives could strengthen food security and align with sustainability goals.

This study aimed to fill these gaps by characterising the rheological and physicochemical properties of *M. sloanei* gum and evaluating the influence of ultrasonication on gum yield and these properties. By improving extraction yield and providing a detailed characterisation of the rheological and physicochemical properties of *M. sloanei* gum, this research aimed to enhance the commercial viability of *M. sloanei* gum for diverse food and pharmaceutical applications, ultimately contributing to more resilient and sustainable food systems.

# 2. Materials and methods

# 2.1. Materials

*Mucuna sloanei* (dehulled) seeds were obtained from the local market in Lagos, Nigeria, and were sorted at 4 °C until dirt and bad seeds were removed. All chemicals and reagents were of analytical grade or the highest available purity and procured from Sigma-Aldrich (St. Louis, MO, USA) or other reputable suppliers.

#### 2.2. Methods

#### 2.2.1. Preparation of Mucuna sloanei flour

The seeds were sorted to remove bad ones before soaking overnight (seeds: distilled water mass ratio 1:10) to soften the seed coat. The seed coat was then removed manually, and the cotyledons were freeze-dried and coarsely milled to a mean particle size of 500  $\mu$ m using an analytical mill (Derskaf Deur, 3100, Sweden). The lipid concentration of the freshweight *M. sloanei* seed flour was reduced by refluxing in 99 % hexane (mass ratio of flour: hexane of 1:3) for two consecutive cycles, followed by filtration and drying to remove excess hexane. The defatted flour was then milled to a finer particle size (mean particle size of 250  $\mu$ m) and stored in an airtight container.

# 2.2.2. Proximate composition of Mucuna sloanei flour

The moisture, ash, and crude fat content of the defatted *M. sloanei* flour were determined according to AOAC methods 925.45B, 942.05, and 920.39A, respectively [15]. Crude protein (calculated as N  $\times$  5.87) was assessed through the Dumas method using the nitrogen combustion system from Gerhardt Dumatherm.

#### 2.2.3. Extraction of Mucuna sloanei gum

Extraction was based on the procedure applied by Brummer et al. (2003) [16] with slight modifications. Briefly, the M. sloanei seed flour was hydrated (mass ratio flour: distilled water of 1:20) at 40 °C while stirring at 900 rpm for 1 h to a fine slurry. The slurry was allowed to cool to 30 °C before ultrasonication (QSONICA 700, Newtown, USA) for 5 mins, with 1-min pulses followed by 1-min rest periods, at 30 % and 60 % amplitudes, resulting in two treatments. The slurry was then centrifuged (HERMLE, Z 366 K, Germany) at 35 °C and 5000 rpm for 20 min. The supernatant was then precipitated by adding 95 % ethanol (mas ratio supernatant: ethanol of 1:2). The hydrocolloid precipitate was pooled together and filtered under suction in a Buchner funnel. The gum was dehydrated in a vacuum at 35  $^\circ \mathrm{C}$  for 15 h resulting in a flaky dried gum, which was easily lifted off the drying tray. The resultant gum was cooled to room temperature in a desiccator. The gum was then pulverised into a fine powder and kept in an airtight container. An unsonicated slurry sample went through the same process and was considered as the control sample in this experiment.

# 2.2.4. Yield and composition of the Mucuna sloanei gum

The yield of gum extracts from *M. sloanei* seed flour was calculated based on *w*/w quantity of gum obtained from 10 g *M. sloanei* seed flour, dry basis, and expressed as a percentage. The moisture and ash content of the gum extract samples were analysed using AOAC methods 925.45B and 942.05, respectively [15]. Crude protein (N  $\times$  5.87) was quantified using the Dumas method with the Gerhardt Dumatherm nitrogen combustion apparatus.

# 2.2.5. Determination of sugar composition in gum

The samples (130 µl) were freeze-dried overnight and then derivatised with 100 µl of methoxyamine at 50 °C for 2 h. Afterwards, 30 µl of BSTFA was added, and derivatisation was conducted at 70 °C for 30 min. The samples were then placed into 2 ml insert vials, and 1 µl was injected into the GC–MS in splitless mode. Separation was performed using a gas chromatograph (6890 N, Agilent Technologies) linked to an Agilent Technologies Inert XL EI/CI Mass Selective Detector (MSD) (5975, Agilent Technologies Inc., Palo Alto, CA). The GC–MS setup was equipped with a CTC Analytics PAL autosampler. Carbohydrates were separated using a non-polar Rxi-5Sil MS capillary column (30 m, 0.25 mm ID, 0.25 µm film thickness). Helium served as the carrier gas, flowing at a rate of 1 ml/min. The injector temperature was set at 250 °C. The temperature program for the oven started at 80 °C for 1 min, followed by a ramping to 300 °C at a rate of 7 °C/min, maintaining this temperature for an additional 2 min [17].

# 2.2.6. Thermal properties of the gum extract

The thermal characteristics of the *M. sloanei* seed flour and extracted gum samples were assessed by applying a high-pressure differential scanning calorimetry (HPDSC) apparatus equipped with STARe software (HPDSC-827, Mettler Toledo, Greifensee, Switzerland). This assessment followed the procedure outlined by Wang et al. (2018) [18]. The melting transformation properties, including onset temperature (To), peak temperature (Tp), conclusion temperature (Tc), and enthalpy ( $\Delta$ H), were recorded for the starches and considered as gelatinisation enthalpy changes.

# 2.2.7. Thermogravimetric analysis

The Thermogravimetric analysis (TGA) of *M. sloanei* flour and gum was carried out using the TA Instruments Thermogravimetric Analyser (Q600 SDT, USA). About 15  $\pm$  0.5 mg samples of similar shape and size were placed in an alumina pan and heated from 25 °C to 600 °C at a rate of 20 °C/min. Each experiment maintained a nitrogen (N2) flow rate of 100 ml/min. Data obtained were evaluated with the aid of TA Universal Analysis software. The thermal degradation onset temperature (5 % weight loss  $T_{5\%}$ ), the temperature at which the highest rate of degradation takes place ( $T_{max}$ ), and the char value (% residue at 600 °C) were determined. The analysis was performed on triplicate samples from a single experiment.

# 2.2.8. Rheological measurements

The Mucuna solutions intended for rheological analysis were created following the procedure outlined by Morales-Contreras et al. (2018) [19] with minor modifications. In summary, dispersions of Mucuna at concentrations of 0.1 %, 0.2 %, 0.5 %, 1 %, 2 %, and 3 % (w/v) were made by combining Mucuna powder with deionised water. These mixtures were then heated and stirred at 40 °C for 5 h. To achieve thorough hydration, the Mucuna solutions were kept overnight in sealed containers at 4 °C to prevent degradation due to bacterial activity. Rheological analyses were performed using a Physica MCR 101 rheometer (Anton Paar, Ostfildern, Germany), utilising a 25 mm crosshatched parallel plate measurement system with a 1 mm gap. Data collection and analysis were performed using Rheoplus software version  $3.0 \times$  (Anton Paar, Ostfildern, Germany). To minimise moisture evaporation during the tests, the external surfaces of the samples were covered with a light coating of paraffin oil. For viscosity measurements, mucuna samples

underwent a steady-shear ramp across a range of shear rates from 0.01 to 1000 s<sup>-1</sup> at 25 °C. This range of shear rates encompasses those commonly used for non-Newtonian fluids during most food processing operations [20]. Linear viscoelastic region (LVR) was investigated using strain sweep tests running across a spectrum of 0.01–1000 % at 6.28 rad/s and 25 °C to select the appropriate strain for viscoelastic analysis. The viscoelastic properties, specifically the storage and loss moduli, were assessed as a function of oscillation frequency through frequency sweep measurements across a range of 0.1 to 150 rad/s, maintaining a constant strain level of 1 % at 25 °C. Temperature sweep was performed in 3 phases, heating from 20 to 90 °C, holding at 90 °C for 10 min, and then colling back to 20 °C at a heating/cooling rate of 5 °C/min. All the rheological analysis was performed on triplicate samples from a single experiment.

# 2.2.9. Statistical analysis

Gums from the triplicate extraction process were used for the experiments described above. One-way analysis of variance (ANOVA) was conducted on the collected data, with comparisons made at p < 0.05 using Fisher's least significant difference (LSD) test. The analysis was carried out using SPSS for Windows version 27 (IBM CA, USA). The independent variables included the raw material and the gums extracted with and without ultrasound assistance, while the dependent variables comprised the measured properties.

# 3. Results and discussion

# 3.1. Chemical composition of Mucuna sloanei flour and extracted gums

The chemical composition of raw M. sloanei seed flour used in this study is presented in Table 1. The moisture content was 17.03  $\pm$  0.8 % (fresh weight), while the fat, protein, and ash contents were 6.16  $\pm$  0.2 %, 23.94  $\pm$  0.22 %, and 3.29  $\pm$  0.24 % (dry basis), respectively. These values are consistent with those reported in previous studies, which found moisture levels between 13 and 17 %, fat at 7.5 %, protein at 22.5 %, and ash at 3.0 % [21]. In comparison, the composition of Mucuna sloanei seeds has been reported to range from 6 to 19 % crude protein, 39.8-61.49 % carbohydrate, 1.84-5.9 % fat, and 11.24-17.10 % vitamins, reflecting the inherent variability in the seed composition [22]. The extracted gum exhibited significantly lower protein (9.99-11.8 %) and ash (0.5-0.59 %) content (Table 2), indicating that the gum is substantially reduced in protein and minerals relative to the cotyledon. These findings are in line with the results reported by Brummer et al. (2003) [16], where a similar extraction process involving 70 % ethanol was used to precipitate water-soluble Fenugreek gum from the supernatant after hydration and centrifugation.

# 3.2. Extraction yield of Mucuna sloanei gum

The results of the investigation into the impact of ultrasound on extraction yield showed that the yield from unsonicated extraction was 41.5 %, while the yields from ultrasonicated assisted extraction (UAE) were 45.81 % and 47.09 % at 30 % and 60 % amplitude applications, respectively (Table 2). This suggests that ultrasonication improved the yield of *M. sloanei* gum by approximately 10.2 % at 30 % amplitude and

### Table 1

Chemical composition (g/100 g) of *Mucuna sloanei* seed flour on fresh weight (FW) and dry basis (db), presented as mean  $\pm$  standard deviation.

Moisture (FW)	Fat (db)	Protein (db)	Ash (db)	*Total CHO (db)	Total Starch (db)	Amylose (% of starch, db)
$\begin{array}{c} 17.03 \pm \\ 0.80 \end{array}$	$\begin{array}{c} 6.16 \\ \pm \ 0.20 \end{array}$	$\begin{array}{c} 23.94 \\ \pm \ 0.22 \end{array}$	$\begin{array}{c} 3.29 \\ \pm \ 0.24 \end{array}$	$\begin{array}{c} 66.54 \\ \pm \ 0.21 \end{array}$	$\begin{array}{c} 1.40 \ \pm \\ 0.11 \end{array}$	$\textbf{0.16} \pm \textbf{0.00}$

CHO = carbohydrates, \*calculated by difference.

#### Table 2

Sample	Energy used (J)	Yield (%, db)	Moisture (%)	Protein (%, db)	Ash (%, db)
US-G SG-30 SG-60	$-9341.0^{b} \pm 17 \\13,877.7^{a} \pm 22$	$\begin{array}{l} 41.57 \pm 1.90^{b} \\ 45.81 \pm 0.22^{a} \\ 47.09 \pm 2.12^{a} \end{array}$	$\begin{array}{l} 5.11 \pm 0.40^{b} \\ 6.65 \pm 0.80^{a} \\ 6.62 \pm 0.41^{a} \end{array}$	$\begin{array}{c} 11.80 \pm 0.41^{a} \\ 9.57 \pm 0.36^{b} \\ 9.99 \pm 0.13^{b} \end{array}$	$\begin{array}{c} 0.51 \pm 0.20^{a} \\ 0.59 \pm 0.04^{a} \\ 0.59 \pm 0.17^{a} \end{array}$

Values that differ in superscript within the same column indicate significant differences (p < 0.05).

US-G: Unsonicated extracted gum; SG-30: Sonication-assisted (at 30 % amplitude) extracted gum; SG-60: Sonication-assisted (at 60 % amplitude) extracted gum.

### 13.3 % at 60 % amplitude, respectively.

Rahman and Lamsal (2021) [23] reviewed the main ultrasonic parameters influencing extraction yield and reported that the rate of extraction or increase in yield during the UAE process could be influenced by operating conditions such as sonic energy density, water-to-flour ratio, agitation speed, and sonication time and temperature. In this experiment, the two amplitude levels (30 % and 60 %) for the UAE were applied under the same operating conditions. Hence, the higher yield observed at the 60 % amplitude UAE could be due to the higher sonication energy density, which induces more intense cavitation. However, the yields obtained at 30 % and 60 % amplitude UAE were not significantly different (p > 0.05) (Table 2).

This finding indicates that the increase in yield using sonication may be enhanced by applying higher amplitudes. The release of gum into solution at respective amplitudes and sonication times could be further enhanced through optimisation of other operating factors and the composition of the flour [24–26].

# 3.3. Sugar composition of Mucuna sloanei flour and extracted gums

The sugar composition of the raw M. sloanei flour and extracted gums, as analysed by HPLC, is presented in Table 3. The analysis identified the presence of a variety of sugars, including L-(+)- arabinose, Dfructose, D-glucose, D-xylose, D-ribose, D-galactose, sorbitol, myoinositol, sucrose, maltose, and trehalose, all in varying proportions in both the raw seed flour and the extracted gums (Table 3). The sugar composition varied significantly (p < 0.05) between the raw *M. sloanei* seed flour and the extracted gums. D-xylose, D-Fructose, L-(+)-Arabinose, and sorbitol concentrations were higher in the unsonicated gum (US-G) compared to the sonicated samples at amplitudes of 30 % (SG-30) and 60 % (SG-60), suggesting that sonication reduced the levels of these sugars. Concentrations of L-(+)-arabinose, D-Ribose, D-fructose, Dglucose, D-Galactose, Myo-Inositol, and Sucrose decreased significantly (p < 0.05) with increasing sonication amplitude. This reduction in sugar concentrations can be attributed to enhanced cavitational forces and thermal effects at higher sonication amplitudes, which may cause disruption of molecular bonds or structural changes in the sugars [27,28].

To the best of our knowledge, no data exist concerning the sugar

#### Table 3

The sugar composition of *Mucuna sloanei* flour and extracted gums presented as mean  $\pm$  standard deviation.

profile of *M. sloanei* gum, making this study the first to report these findings. The sugar concentrations observed here are consistent with those reported for similar seed gums, including fenugreek, flaxseed, yellow mustard, and psyllium gums [16,29].

### 3.4. Thermal properties of Mucuna sloanei flour and extracted gums

The differential scanning calorimetry (DSC) thermogram parameters for *M. sloanei* gums and the raw flour are presented in Table 4. The gelatinisation endotherms, characterised by onset (To), peak (Tp), and conclusion (Tc) temperatures, were 92.12, 97.24, and 103.40 °C, for raw *M. sloanei*, respectively. Notably, no significant variation was observed (p > 0.05) in the gelatinisation temperature range, defined by To and Tc, among the raw flour (92.12 and 103.40 °C) and the extracted gums, unsonicated (US-G) (93.49 and 103.40 °C), sonication-assisted at 30 % amplitude (SG-30) (90.14 and 100.00 °C), and sonication-assisted at 60 % amplitude (SG-60) (92.22 and 100.00 °C), as detailed in Table 4.

These findings differed from the lower To, Tp, and Tc temperatures of 29.52, 45.45, and 98.0 °C, respectively, reported by Uzomah and Odusanya (2011) [2] for the defatted *M. sloanei* flour. The differences may be attributed to variations in DSC equipment, sample weight, and heating rates employed in different studies. Furthermore, the gelatinisation temperatures observed in this research are markedly higher than those reported for various starches, as reviewed by Hoover and Sosulski (1991) [30]. This can be attributed to the notably low starch content (1.4 %) in raw *M. sloanei* (see Table 1) in the current study, likely due to a substantial portion of the starch being sedimented during the centrifugation step of the gum extraction process.

The melting enthalpy ( $\Delta$ H) of raw *M. sloanei* flour was significantly greater (p < 0.05) than the extracted gums, potentially due to its higher protein content (see Table 1). The interactions between proteins and polysaccharides likely result in the development of soluble complexes stabilised by electrostatic interactions and hydrogen bonding. The higher ratio of disulfide bonds (DSB) to hydrogen bonds (HB) in the raw flour may also account for the increased thermal energy required to melt the sample [31].

Sugars mg/L(ppm)	Sample					
	RM	US-G	SG-30	SG-60		
D-Xylose	$0.466\pm0.28^{\rm b}$	$0.655\pm0.03^{\rm a}$	$0.181\pm0.13^{\rm c}$	$0.171\pm0.11^{\rm c}$		
L-(+)-Arabinose	$3.350\pm0.16^{a}$	$2.759\pm0.23^{\rm b}$	$0.906\pm0.08^{\rm c}$	$0.306\pm0.05^d$		
D-Ribose	$0.608\pm0.40^{a}$	$0.079\pm0.04^{\rm d}$	$0.423\pm0.27^{\rm b}$	$0.256\pm0.10^{\rm c}$		
D-Fructose	$28.73\pm0.12^{\rm a}$	$18.02\pm0.01^{\rm b}$	$14.78\pm0.02^{\rm c}$	$5.43\pm0.14^{\rm d}$		
D-Galactose	$31.33\pm0.10^{\rm b}$	$20.13\pm0.30^{\rm c}$	$40.92\pm0.07^a$	$5.53\pm0.20^{\rm d}$		
D-Glucose	$32.37\pm0.17^{\rm a}$	$20.25\pm0.32^{\rm b}$	$18.07\pm0.25^{\rm b}$	$4.35\pm0.39^{\rm c}$		
Sorbitol	$2.89\pm0.00^{\rm b}$	$3.95\pm0.12^{\rm a}$	$0.29\pm0.12^{\rm c}$	$0.38\pm0.26^{\rm c}$		
Myo-Inositol	$14.98\pm0.32^{\rm ab}$	$\textbf{7.45}\pm0.20^{\rm c}$	$17.67\pm0.03^{\rm a}$	$12.61\pm0.02^{\rm b}$		
Sucrose	$95.53\pm0.10^{\rm a}$	$79.28\pm0.03^{\rm b}$	$119.56 \pm 0.37^{a}$	$77.41 \pm 0.32^{\mathrm{b}}$		
Maltose and Trehalose	$8.50\pm0.08^{\rm c}$	$4.62\pm0.05^{\rm b}$	$4.73\pm0.01^{\rm b}$	$2.72\pm0.07^{\rm a}$		

Values that differ in superscript within the same row indicate significant differences (p < 0.05).

RM: Raw Mucuna sloanei flour; US-G: Unsonicated gum extract; SG-30: Sonication-assisted (30 % amplitude) extracted gum; SG-60: Sonication-assisted (60 % amplitude) extracted gum.

Table 4

Thermal characteristics of Mucuna sloanei flour and extracted gums.

Sample	Onset (°C)	Peak (°C)	Conclusion (°C)	Range	$\Delta H (J/g)$
RM	$92.12\pm2.71^{\rm a}$	$97.24\pm2.80^{\rm a}$	$103.40\pm0.16^{\rm a}$	$11.28\pm2.80^{\rm a}$	$2.40\pm0.53^{a}$
US-G	$93.49 \pm 1.16^{\rm a}$	$97.70 \pm 0.41^{a}$	$103.40 \pm 1.40^{\rm a}$	$10.00\pm0.36^{\rm a}$	$1.25\pm0.18^{ab}$
SG-30	$90.14 \pm 1.00^{\rm a}$	$96.00\pm3.14^{a}$	$100.00 \pm 3.42^{a}$	$10.50\pm2.71^{a}$	$1.56\pm0.43^{ab}$
SG-60	$92.22\pm1.91^{\rm a}$	$96.00 \pm 1.25^{\text{a}}$	$100.00\pm0.35^a$	$8.00\pm1.52^{\text{a}}$	$1.07\pm0.04^{b}$

Values that differ in superscript within the same column indicate significant differences (p < 0.05).

RM: Raw Mucuna sloanei flour; US-G: Unsonicated gum extract; SG-30: Sonication-assisted (30 % amplitude) extracted gum; SG-60: Sonication-assisted (60 % amplitude) extracted gum.

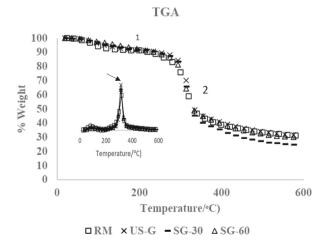
# 3.5. Thermogravimetric properties of Mucuna sloanei flour and gums

Thermogravimetric analysis (TGA) provides insight into the heat stability and degradation behaviour of materials by assessing mass loss in relation to temperature. In hydrocolloids, TGA is particularly useful in assessing heat-induced bond dissociation due to thermal energy surpassing bond strength [32]. The TGA and derivative TGA (DTGA) curves for *M. sloanei* flour and the extracted gums are presented in Fig. 1, and the key thermogravimetric parameters are summarised in Table 5.

Two distinct mass loss events (1 and 2) were observed in the thermogravimetric curve (Fig. 1). This pattern is consistent with the research by Alpizar-Reyes et al. (2017) [33], which stated two similar mass loss stages in tamarind (*Tamarindus indica* L.) seed mucilage. The initial mass loss of about 5 % occurred at temperatures exceeding 75 °C, likely representing the evaporation of free water within the hydrocolloid matrix. This initial phase could be attributed to the hydrophilic traits of polysaccharide functional groups, facilitating moisture evaporation [32,34].

The degradation onset temperature ( $T_{5\%}$ ) was notably (p < 0.05) higher in the extracted gums compared to the raw *M. sloanei* flour (RM), indicating improved thermal stability in the gums. This finding aligns with the results reported by Attama and Akpa (2008), who observed similar improvements in thermal properties after gum extraction [35]. This increased stability may be a result of the processes of gum extraction and sonication, which enhance polymer interactions by altering molecular structures [33]. However, no notable variations (p > 0.05) were detected among the samples with respect to maximum degradation temperature ( $T_{max}$ ), as shown in Table 5.

The second mass loss event, which occurred at temperatures higher than 300 °C, corresponded to a residue percentage at  $T_{max}$  ranging from 61.79 % to 65.05 %, indicating mass losses of 38.21 % and 34.95 %, respectively. These findings suggest that raw *M. sloanei* flour (RM)



**Fig. 1.** Thermogravimetric curves showing the thermal properties of raw *Mucuna sloanei* flour (RM), unsonicated extracted gum (US-G), sonication-assisted (at 30 % amplitude) extracted gum (SG-30), and sonication-assisted (at 60 % amplitude) extracted gum (SG-60).

# Table 5

Thermogravimetric parameters, including degradation onset temperature ( $T_{5\%}$ ), maximum degradation temperature ( $T_{max}$ ), percentage residue at  $T_{max}$ , and percentage residue at 600 °C, of *Mucuna sloanei* flour and extracted gums.

Sample	T <sub>5%</sub>	T <sub>max</sub>	% Residue at T <sub>max</sub>	% Residue at 600 °C
RM	$94.21\pm0.10^{d}$	$317.46\pm0.03^a$	$61.79 \pm 0.02^d$	$30.87 \pm 0.00^a$
US-G	$117.61 \pm 0.06^{c}$	$317.58 \pm 0.01^{\rm a}$	$65.05\pm0.08^{\rm a}$	$\mathbf{31.02^a} \pm \mathbf{0.01^a}$
SG-30	$130.40\pm0.04^{\mathrm{b}}$	$316.37 \pm 0.01^{a}$	$63.87\pm0.02^{\rm b}$	$25.55\pm1.43^{\rm b}$
SG-60	$146.87\pm0.00^a$	$315.50\pm3.02^a$	$62.95 \pm \mathbf{3.60^c}$	$29.03 \pm 0.81^a$

Values that differ in superscript within the same column indicate significant differences (p < 0.05).

RM: Raw *Mucuna sloanei* flour; US-G: Unsonicated gum extract; SG-30: Sonication-assisted (at 30 % amplitude) extracted gum; SG-60: Sonication-assisted (at 60 % amplitude) extracted gum.

exhibited the highest mass loss (38.21 %), while unsonicated extracted gum (US-G) showed the lowest mass loss (34.95 %). This could be attributed to the higher purity of the extracted gum compared to the raw flour, which leads to reduced decomposition of non-gum components, such as lipids, during thermal analysis [36]. The sonication-assisted extractions, specifically SG-30 and SG-60, exhibited residue percentages at  $T_{max}$  of 63.87 % and 62.95 %, corresponding to mass losses of 36.13 % and 37.05 %, respectively. This indicates that their mass loss is slightly higher than that of the unsonicated extract (US-G) at 34.95 %. The enhanced decomposition in sonicated samples might be explained by the collapse of cavitational bubbles during the sonication process, which disrupts molecular bonds and results in reduced thermal stability [11,24]. These findings suggest that the sonication process could significantly impact the structural integrity of the hydrocolloids, thereby altering their thermal behaviour.

The thermal and thermogravimetric properties of *Mucuna sloanei* flour and gums presented in this study, including gelatinisation temperatures and degradation profiles, offer key insights into their thermal stability and processing characteristics. These characteristics are crucial for their applications in sustainable food reformulation, controlled-release delivery systems, and pharmaceutical formulations, helping to better link structure to functionality across diverse uses.

# 3.6. Rheological properties of Mucuna slonei flour and extracted gums

### 3.6.1. Flow behaviour of Mucuna sloanei flour and extracted gums

The shear stress in relation to the shear rate for *M. sloanei* flour and extracted gums is shown in Fig. 2. The flow behaviour curves exhibit typical shear-thinning behaviour, where shear stress increases with the shear rate. This behaviour is characteristic of random coil polymers, where the movement of dispersed molecules under shear induces internal resistance [37]. Typical shear-thinning behaviour observed for *M. sloanei* flour and extracted gums was consistent with other hydrocolloids, such as carrageenan and xanthan gum studied by Marcotte et al. (2001) [38].

The yield stress, which refers to the minimum stress required for the material to begin flowing, was lower in raw *M. sloanei* flour compared to the isolated gums (Fig. 2), suggesting a less coherent network in the flour

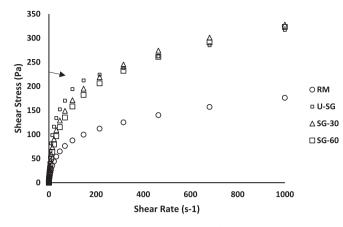


Fig. 2. Flow behaviour of raw *Mucuna sloanei* flour (RM), unsonicated gum extract (US-G), and sonication-assisted gum extracts at 30 % (SG-30) and 60 % (SG-60) amplitude, in 3 % aqueous solution at 25  $^{\circ}$ C, showing the correlation between shear stress and shear rate.

which could be due to its heterogeneous composition (see Table 1). In contrast, the isolated gums exhibited greater internal stress development due to a more uniform and structured polymer network. The ultrasonication process appeared to reduce the yield stress of the extracted gums, evidenced by the lower yield points of SG-30 and SG-60 compared to unsonicated gums (US-G) shown by the arrow in Fig. 2. This is likely due to cavitation effects during ultrasonication, which disrupted intermolecular forces and reduced chain entanglement, leading to weaker overall structural integrity [27,28].

The apparent viscosity of aqueous solutions of *M. sloanei* flour and gums at concentrations of 1 %, 2 %, and 3 % are illustrated in Fig. 3. At lower shear rates, all concentrations exhibited a more Newtonian behaviour, transitioning to more pronounced shear-thinning behaviour

at elevated shear rates. Additionally, increased concentrations resulted in higher viscosity, with the shear-thinning effect becoming more evident. Experimental data were best fitted with the Power-law (Ostwald-de Waele) model (Eq. (1)), yielding optimal results specifically for the 3 % polymer concentration, as presented in Table 6.

$$\sigma = K.\gamma^n \tag{1}$$

where  $\sigma$  and  $\gamma$  represent the shear stress (Pa) and shear rate (s<sup>-1</sup>), respectively, *K* denotes the consistency coefficient (Pa.s<sup>n</sup>) and *n* indicates the flow behaviour index. The extracted gums demonstrated considerably greater viscosity (*P* < 0.05) than the *M. sloanei* flour at both shear rates of 0 ( $\eta_0$ ) and 100 ( $\eta_{100}$ ) s<sup>-1</sup> (Table 6). Furthermore, no meaningful variation (*P* > 0.05) was observed in the flow behaviour

# Table 6

Apparent viscosity at 0 and 100 s<sup>-1</sup> shear rates, along with the parameters (*K*,n) of the Power-law model for *Mucuna sloanei* flour and gum solutions (3 % concentration) at 25 °C.

	Viscosity (Pa.s)			Power law parameters	
	At 0 s <sup>-1</sup> shear rate ( $\eta_0$ )	At 100 s <sup><math>-1</math></sup> shear rate ( $\eta_{100}$ )	<i>n</i> -value	K (Pa.s <sup>n</sup> )	R <sup>2</sup>
RM	$5.24\pm0.0^{c}$	$0.78\pm0.1^{b}$	$0.55 \pm 0.0^{\mathrm{a}}$	$\begin{array}{c} 6.08 \pm \\ 0.1^{b} \end{array}$	$\begin{array}{c} 0.95 \pm \\ 0.0^{a} \end{array}$
US- G	$25.10\pm0.6^a$	$1.94\pm0.0^a$	$\begin{array}{c} 0.67 \pm \\ 0.0^{a} \end{array}$	$\begin{array}{c} 10.67^{\mathrm{b}} \pm \\ 0.5^{\mathrm{a}} \end{array}$	$\begin{array}{c} \textbf{0.86} \ \pm \\ \textbf{0.0^b} \end{array}$
SG- 30	$13.23\pm0.3^{b}$	$1.74\pm0.0^{a}$	$0.71~\pm$ $0.0^{\mathrm{a}}$	$6.77~\pm$ $0.1^{ m b}$	$\begin{array}{c} 0.87 \pm \\ 0.0^{b} \end{array}$
SG- 60	$12.35\pm0.1^{b}$	$1.43\pm0.2^a$	$\begin{array}{c} 0.70 \ \pm \\ 0.0^a \end{array}$	$\begin{array}{c} 6.01 \ \pm \\ 0.0^b \end{array}$	$\begin{array}{c} \textbf{0.88} \pm \\ \textbf{0.0^b} \end{array}$

Values that differ in superscript within the same column indicate significant differences (p < 0.05).

RM: Raw *Mucuna sloanei* flour; US-G: Unsonicated gum extract; SG-30: Sonication-assisted (at 30 % amplitude) extracted gum; SG-60: Sonication-assisted (at 60 % amplitude) extracted gum.

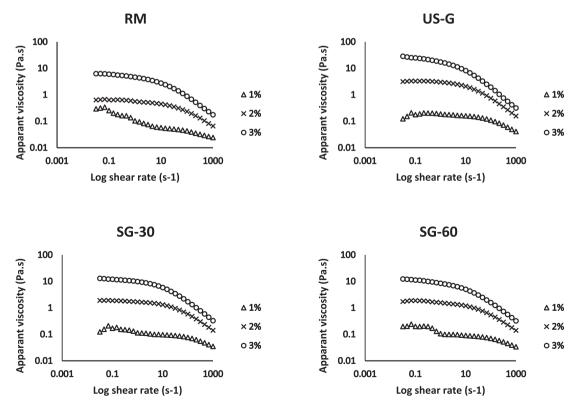


Fig. 3. Apparent viscosity of raw *Mucuna sloanei* flour (RM), unsonicated gum extract (US-G), and sonication-assisted gum extracts at 30 % (SG-30) and 60 % (SG-60) amplitude at concentrations of 1 %, 2 %, and 3 % versus shear rates ranging from 0.01 to 1000 s<sup>-1</sup> measured at 25 °C.

index among the flour, unsonicated gum, and sonicated gums at 30 % and 60 % amplitude (Table 6). This suggests that the microstructural rearrangement within the range of applied shear was not significantly influenced by sonication and its parameters.

Fig. 3 illustrates the apparent viscosity of raw M. sloanei flour and the gums at concentrations of 1 %, 2 %, and 3 % versus shear rates ranging from 0.01 to 1000 s<sup>-1</sup> measured at 25 °C. The apparent viscosity of samples exhibited a tendency to stabilise at low shear rates, termed zero shear viscosity [39]. Above a critical shear rate, the samples exhibited shear-thinning behaviour, characterised by decreasing viscosity with increasing shear rates. These shear-thinning characteristics have been observed in similar studies for various hydrocolloids, such as carrageenan and xanthan gums [38] as well as pectin [40]. In biopolymers, shear-thinning behaviour is often attributed to microstructural and molecular structure breakdown [41]. The results from this study indicated that *M. sloanei* flour and gums are low in starch but relatively high in protein and non-starch polysaccharides (Table 1 and 2). According to Sadeghi et al. (2021) [42], the viscosity in protein-polysaccharide systems can be enhanced through interactions between proteins and polysaccharides, stabilised through electrostatic interactions and hydrogen bonding, facilitating intra- and interchain interactions. This suggests that the alignment of swollen micro-molecules within proteinpolysaccharide co-polymer chains under shear may contribute to the observed shear-thinning behaviour (Table 6, Fig. 3).

Despite the application of sonication, the shear-thinning behaviour of the gums remained largely unaffected, indicating the structural robustness of the *M. sloanei* gum (Table 6). This resilience suggests that the polymer network can withstand mechanical processing without significant alteration in its flow behaviour, making *M. sloanei* gum a promising candidate for various food industrial applications. These findings highlight the versatility of *M. sloanei* gum in food products, where maintaining consistent rheological properties under different processing conditions is critical. Furthermore, the ultrasonication treatment may offer a potential pathway to fine-tune other structural aspects of the gum without compromising its critical flow properties.

#### 3.6.2. Viscoelastic behaviour of Mucuna sloanei flour and extracted gums

The amplitude sweep test was conducted to determine the linear viscoelastic region (LVR) of the samples, ensuring that all viscoelastic characteristics were analysed within this range to prevent structural breakdown. Fig. 4 illustrates the variations in storage modulus (G') and loss modulus (G") for the aqueous solutions of the samples at a concentration of 3 % across a temperature range of 20-90 °C (temperature sweep test) and during a time-dependent analysis of G' and G" at 90 °C (time sweep test). The focus on a 3 % concentration for viscoelastic assessment arose from preliminary analyses indicating it provided distinct viscoelastic characteristics between samples compared to 1 % and 2 %. This minimised variability, allowing for better insight into the structural integrity and functional properties of *M. sloanei* flour and gum for potential industrial applications.

Notably, as temperature increased, the gum exhibited an initial predominantly viscous behaviour (G' > G'), transitioning to a predominantly elastic response (G' > G') at higher temperatures. This transition was observed at 90 °C, though at different time intervals for the various samples. For all samples, there was an initial slight decrease in both G' and G''. Subsequently, the moduli increased sharply with rising temperature and during the cooling phase. The elastic modulus demonstrated a steeper slope, indicating enhanced elastic behaviour of the gums when maintained at elevated temperatures for extended periods (Fig. 4).

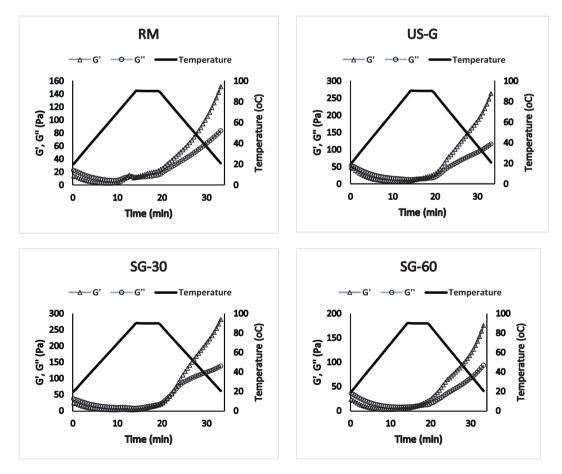


Fig. 4. Storage and loss modulus of raw *Mucuna sloanei* flour (RM), unsonicated gum extract (US-G), and sonication-assisted gum extracts at 30 % (SG-30) and 60 % (SG-60) amplitude at concentrations of 3 % as a function of temperature, 20–90 °C, and time.

The continuous increase in both elastic and loss moduli during the cooling phase suggests the formation of a reinforced gel at lower temperatures. Furthermore, the plateau observed for G' and G" during the holding period at 90 °C reflects the thermal stability of the gels under high-temperature conditions [43]. These findings provide critical insights into the rheological characteristics and potential functional applications of *M. sloanei* gum in industrial contexts.

The variation in the storage (G') and loss (G") moduli for the samples at 3 % concentration in aqueous solutions and a constant strain of 1 % across a frequency range of 0.1 to 100 rad/s at 25 °C is presented in Fig. 5. In the lower ranges of angular frequencies, the gum exhibited primarily viscous behaviour (G" > G'), transitioning to a more elastic response (G' > G") at higher frequencies. Notably, the unsonicated gum demonstrated greater frequency dependence than the sonicated samples, with higher values for both G' and G" throughout the frequency range. This suggests a greater capacity for the unsonicated gum to form firmer gels with enhanced stiffness [43].

The crossover point (gelation point) observed in the ultrasoundassisted extracted gums occurred at higher frequencies than in the unsonicated extracted gum, indicating lower reaction kinetics (slower gelation process) for the ultrasound-assisted samples [43]. This finding suggests that the ultrasound treatment may enhance the structural integrity and stability of the gum at elevated frequencies, making it more suitable for specific applications in food and pharmaceutical industries. Additionally, ultrasound treatment can induce structural changes by disrupting some crosslinks in the gel macromolecules [44]. These structural modifications may improve the stability and texture of food products by enabling better control over the rheological behaviour during processing.

### 3.6.3. Principal component analysis

Principal Component Analysis (PCA) of the data (Fig. 6) highlighted the influence of sonication energy on the key physicochemical and thermal properties of the samples. The first two principal components contributed to 75 % of the variance observed between raw *M. sloanei* flour and the isolated gums (Fig. 6a). The sonicated samples (SG-30 and SG-60) clustered distinctly in the same quadrant, separating them clearly from the raw flour and unsonicated gum (US-G). These sonicated samples exhibited significant positive correlations (p < 0.05) with sonication energy and degradation onset temperature (T5%) while demonstrating significant negative correlations with raw *M. sloanei* flour. These findings suggest that the applied sonication energy substantially influenced both gum extraction efficiency from *M. sloanei* and the thermal stability of the extracted gums at T5%.

The results from Thermogravimetric Analysis (TGA) (Fig. 1) further support the PCA findings by revealing two distinct weight loss regions. The first degradation phase, attributed to moisture loss [36,45], indicated that the raw *M. sloanei* exhibited moisture loss at significantly lower temperatures (p < 0.05) in comparison with the isolated gums. This difference likely arises from heterogeneous composition and weaker intermolecular forces in raw *M. sloanei*, which fail to retain water molecules effectively. In contrast, the enhanced thermal stability observed in the sonicated gums (p < 0.05) during the initial thermal transition suggested that sonication improved gum integrity, increasing its resistance to moisture-related thermal degradation [46,47].

The PCA biplot (Fig. 6a) further demonstrated a robust positive correlation between the raw *M. sloanei* flour and its thermal and thermogravimetric properties, implying that these variables are key in explaining the differences observed between the samples. Additionally, viscosity parameters exhibited negative correlations with most sugar and proximate compositions of the samples. The heterogeneous nature of raw *M. sloanei* flour likely contributed to its lower viscosity at both low and high shear rates compared to the more homogeneous structure of the isolated gums (Table 6).

The analysis of the first and third principal components (Fig. 6b), which collectively explained 63 % of the variation, revealed notable similarities between the unsonicated gum (US-G) and the gum extracted with 30 % sonication amplitude. Both samples were positively correlated with viscosity parameters, suggesting that 30 % sonication amplitude may not induce sufficient structural disruption to significantly alter the rheological characteristics of the gum.

Furthermore, the raw M. sloanei flour exhibited a more significant

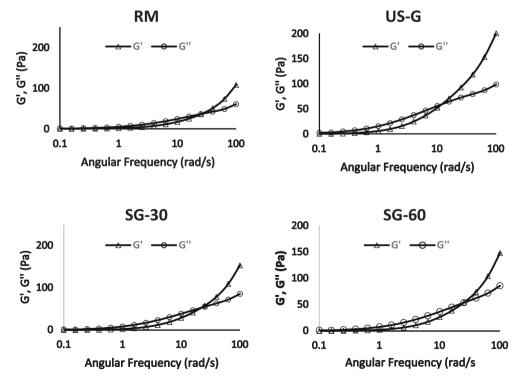
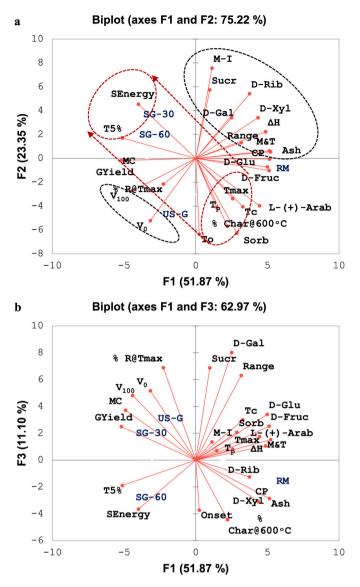


Fig. 5. Storage and loss modulus of raw *Mucuna sloanei* flour (RM), unsonicated gum extract (US-G), and sonication-assisted gum extracts at 30 % (SG-30) and 60 % (SG-60) amplitude, at a concentration of 3 %, as a function of frequency (0.1–100 rad/s).



**Fig. 6.** PCA biplot of assessed characteristics demonstrating the similarities and differences between the samples and the key variables contributing to these variations. Variables include moisture content (MC), crude protein (CP), ash, sonication energy (SEnergy), gum yield (GYield), onset temperature (To), peak temperature (Tp), concluding temperature (Tc), enthalpy change ( $\Delta$ H), and various sugars, including D-Xylose (D-Xyl), L-(+)-Arabinose (L-(+)-Arab), D-Ribose (D-Rib), D-Fructose (D-Fruc), D-Galactose (D-Gal), D-Glucose (D-Glu), sorbitol (Sorb), myo-inositol (M-I), sucrose (Sucr), maltose and trehalose (M&T). Additionally, the plot shows percentage residue at maximum temperature (Tmax), degradation onset temperature (T5%), and viscosity at zero (V<sub>0</sub>) and at 100 s<sup>-1</sup> shear rates (V<sub>100</sub>).

shear-thinning behaviour than the isolated gums, potentially due to its heterogeneous composition. In contrast, the sonicated gums, characterised by relatively homogeneous protein-polysaccharide copolymer chains, displayed greater resistance to shear-thinning (Table 6). This enhancement may arise from the reinforcement of polymer chains during shear, a phenomenon less prominent in the raw material and unsonicated gum. Sonication likely facilitated the removal of fibrous material prior to extraction, resulting in a more homogeneous gum compared to raw *M. sloanei* flour and unsonicated gum [48]. While no correlation was observed between sugar composition and rheological properties, sugar content showed positive correlations with thermal properties (Fig. 6). This aligns with Salehi et al. (2018) [48], who reported that thermal properties are significantly influenced by material composition and molecular structure.

The PCA biplot, highlighted the similarities and differences among the samples while identifying the key variables that drive these variations. The findings from this study provide novel insight into the influence of sonication on the physicochemical, rheological and thermal behaviour of *M. sloanei* gums, which can have important implications for their application in food processing and quality assurance.

In summary, sonication significantly influenced the physicochemical, thermal, and rheological properties of *Mucuna sloanei* extracted gums. The sonicated gums showed improved extraction yield, thermal stability, and viscosity compared to the raw flour, with the 60 % amplitude treatment producing the most pronounced effects. Although no significant differences in gelatinisation temperatures were observed, sonication enhanced the thermal stability of the gums, likely by improving their structural integrity. Furthermore, sonication altered the flow properties, reducing yield stress and shear-thinning behaviour of the gums. The principal component analysis further validated that sonication positively influenced the physicochemical and rheological characteristics of gums, reinforcing their potential as a promising ingredient for various food and industrial applications.

While this study demonstrated the beneficial effects of sonication on *Mucuna sloanei* gum extraction yield and its physicochemical and rheological properties, certain limitations remain. This study primarily examined the immediate impact of sonication, without assessing the long-term stability or shelf-life of the sonicated gums, factors critical for their practical applications. Additionally, the study did not evaluate the performance of these gums in model food products and drug delivery systems, leaving uncertainties about their functionality under real processing conditions. Future research should address these aspects to provide a more comprehensive understanding of their industrial applicability.

# 4. Conclusion

The primary objective of the study was to examine the effects of sonication at amplitudes of 30 % and 60 % on extraction efficiency, as well as the physicochemical and rheological properties of Mucuna sloanei gum, with the broader aim of improving the functionality of gum for industrial applications. This study represents the first comprehensive analysis of the sugar profile, as well as detailed rheological and thermal behaviour of M. sloanei gum. Chemical composition analysis revealed that raw M. sloanei flour contained 17.03 % moisture, 6.16 % fat, 23.94 % protein, and 3.29 % ash, while the extracted gums exhibited significantly lower protein content (10.0-11.8 %) and ash (0.5-0.6 %), suggesting fewer non-gum components and a more refined product. The sonication process significantly enhanced extraction yields, with values reaching 45.81 % and 47.09 % for 30 % and 60 % amplitude, respectively, compared to 41.5 % for unsonicated extraction. These results reflect increases of approximately 10.2 % and 13.3 % in the extraction efficiency, directly correlating with the amplitude of sonication. Sonication also altered sugar concentrations, reducing levels of D-xylose, Dfructose, L-(+)-arabinose, and sorbitol. These changes in sugar composition were statistically significant (p < 0.05) and may influence the gum functionality in food and pharmaceutical formulations. The reduction of certain sugars, in particular, could affect the gum solubility and gelling properties, offering insights into its modified functional characteristics after sonication. Thermal analysis revealed no significant differences (p > 0.05) in gelatinisation temperatures among gum samples, suggesting that sonication enhanced yields without compromising thermal stability, a promising outcome for processing applications, where heat stability is critical. Thermogravimetric analysis (TGA) demonstrated that extracted gums had higher degradation onset temperatures (T5%) than raw flour. This finding supports the hypothesis that sonication enhanced the thermal stability of M. sloanei gum, making it more suitable for industrial applications that require resistance to high temperatures. The

viscoelastic analysis further confirmed that the extracted gums had higher yield stress values than M. sloanei flour, indicating superior binding and thickening performance, making them suitable as functional ingredients in food and pharmaceutical products. Additionally, the extracted gums displayed notable shear-thinning behaviour and robust viscoelastic characteristics, with unsonicated gum showing greater stiffness and gel formation capacity. Principal Component Analysis (PCA) indicated that sonication played a key role in modulating the physicochemical and thermal properties of the gum. Sonicated samples (SG-30 and SG-60) were positively correlated with degradation onset temperature (T5%), suggesting that sonication effectively improved gum stability. In summary, this study demonstrated the potential of M. sloanei gum as a functional ingredient for food and pharmaceutical industries and highlighted how optimised sonication conditions could improve yield and desired physicochemical and rheological characteristics. Addressing the gap in knowledge regarding M. sloanei as an underutilised African indigenous crop is crucial, particularly in regions reliant on imported, unsustainable resources. Developing local alternatives could strengthen food security and align with sustainability goals, making this study a significant step towards enhancing its industrial potential.

# CRediT authorship contribution statement

Adedola S. Adeboye: Writing – original draft, Visualization, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. Atefeh Amiri-Rigi: Writing – review & editing, Writing – original draft, Validation, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. Naushad M. Emmambux: Writing – review & editing, Validation, Supervision, Resources, Project administration, Funding acquisition, Data curation.

## Funding

This work was supported by the DSI -NRF Centre of Excellence in Food Security [grant number 91490]. The funder had no role in the study design, data collection, analysis, interpretation, writing of the report, or decision to submit the article for publication.

# Declaration of competing interest

We have nothing to declare.

# Data availability

Data will be made available on request.

#### References

- Y. Ga, R. Liu, H. Liang, Food hydrocolloids: structure, properties, and applications, Foods 13 (7) (2024) e1077, https://doi.org/10.3390/foods13071077.
- [2] A. Uzomah, O.S. Odusanya, Mucuna sloanei, Detarium microcarpum and Brachystegia eurycoma seeds: preliminary study of their starch-hydrocolloid system, Afr. J. Food Sci. 5 (13) (2011) 733–740, https://doi.org/10.5897/ AJFS11.088.
- [3] I.A. Ajayi, R.A. Oderinde, D.O. Kajogbola, J.I. Uponi, Oil content and fatty acid composition of some underutilized legumes from Nigeria, Food Chem. 99 (1) (2006) 115–120, https://doi.org/10.1016/j.foodchem.2005.06.045.
- [4] G.C. Ugwu, V.C. Ejere, C.L. Okanya, J.N. Omeje, J.V. Egbuji, M.C. Onu, C. O. Chukwuka, Effects of aqueous seed extracts of *Mucuna sloanei* (Fabaceae) on body weight and some biochemical parameters of *Rattus novergicus*, Afr. J. Biotechnol. 17 (28) (2018) 885–891, https://doi.org/10.5897/AJB2017.16112.
- [5] C.E. Ugwu, F.C. Kenechukwu, E.O. Diovu, H.O. Udodeme, M.A. Momoh, E. B. Onuigbo, A.A. Attama, Exploitation of capsule system for colon targeted drug delivery of biopolymer-based microparticles: *in vivo* and *in vitro* applications, Heliyon 8 (11) (2022) e11390, https://doi.org/10.1016/j.heliyon.2022.e11390.
- [6] A.A. Attama, O.J. Nwabunze, Mucuna gum microspheres for oral delivery of glibenclamide: *in vitro* evaluation, Acta Pharma. 57 (2) (2007) 161–171, https:// doi.org/10.2478/v10007-007-0013-9.
- [7] J. Eke-Ejiofor, C.U. Awajiogak, Effect of processing methods on the physicochemical, antinutrient and pasting properties of three commonly consumed

soup thickeners in Nigeria, Asian Food Sci. J. 13 (4) (2019) 1–16, https://doi.org/ 10.9734/afsj/2019/v13i430115.

- [8] F.Y. Oladipo, C.E. Chigbundu, V.N. Atasie, I. Iyeimo, Extraction, purification and analysis of Mucuna sloanei seed gum, J. Appl. Res. Technol. 94 (2) (2013) 94–100.
- [9] J.C. Onweluzo, Z.A. Obanu, M.C. Okwandu, Potentials of gum from *Detarium microcarpum* (DM) and *Mucuna flagellipes* (MF) seeds as raw beef burger stabilizers, Plant Foods Hum. Nutr. 59 (4) (2004), https://doi.org/10.1007/s11130-004-0027-0.
- B.K. Tiwari, K. Muthukumarappan, C.P. O'donnell, P.J. Cullen, Rheological properties of sonicated guar, xanthan and pectin dispersions, Int. J. Food Prop. 13 (2) (2010) 223–233, https://doi.org/10.1080/10942910802317610.
- [11] M. Nowak, P. Szperlich, E. Talik, J. Szala, T. Rzychoń, D. Stróż, A. Nowrot, B. Solecka, Sonochemical preparation of antimony subiodide, Ultrason. Sonochem. 17 (1) (2010) 219–227, https://doi.org/10.1016/j.ultsonch.2009.05.016.
- [12] A. Amiri-Rigi, S. Abbasi, M.G. Scanlonb, Enhanced lycopene extraction from tomato industrial waste using microemulsion technique: optimization of enzymatic and ultrasound pre-treatments, Innov. Food Sci. Emerg. Technol. 35 (2016) 160–167, https://doi.org/10.1016/j.ifset.2016.05.004.
- [13] K. Ashley, R.N. Andrews, L. Cavazos, M. Demange, Ultrasonic extraction as a sample preparation technique for elemental analysis by atomic spectrometry, J. Anal. At. Spectrom. 16 (10) (2001) 1147–1153, https://doi.org/10.1039/ B102027G.
- [14] L.M. Nwokocha, P.A. Williams, Isolation and rheological characterization of Mucuna flagellipes seed gum, Food Hydrocoll., 23 (5) (2009), 1394–1397. t: https ://wrexham.repository.guildhe.ac.uk/id/eprint/248.
- [15] Official methods of analysis of AOAC International, in: H. Williams (Ed.), Method, 17th ed., 2000, 925.45B, 942.05 and 920.39A, AOAC, Gaithersburg, MD, USA.
- [16] Y. Brummer, W. Cui, Q. Wang, Extraction, purification and physicochemical characterization of fenugreek gum, Food Hydrocoll. 17 (3) (2003) 229–236, https://doi.org/10.1016/S0268-005X(02)00054-1.
- [17] S.N. Walford, GC-MS as a tool for carbohydrate analysis in a research environment, Proc. Int. Soc. Sugarcane Technol. 27 (2010) 1–15. https://api.semanticscholar.or g/CorpusID:53870232.
- [18] S. Wang, C. Chao, F. Xiang, X. Zhang, S. Wang, L. Copeland, New insights into gelatinization mechanisms of cereal endosperm starches, Sci. Rep. 8 (2018) e3011, https://doi.org/10.1038/s41598-018-21451-5.
- [19] B.E. Morales-Contreras, W. Rosas-Flores, J.C. Contreras-Esquivel, L. Wicker, J. Morales-Castro, Pectin from husk tomato (Physalis ixocarpa Brot.): rheological behavior at different extraction conditions, Carbohydr. Polym. 179 (2018) 282–289, https://doi.org/10.1016/j.carbool.2017.09.097.
- [20] G. Mazza, C.G. Biliaderis, Functional properties of flax seed mucilage, J. Food Sci. 54 (2006) 1302–1305, https://doi.org/10.1111/j.1365-2621.1989.tb05978.x.
- [21] M. Dosunmu, E. Ekerendu, The chemical composition and physico-chemical properties of Mucuna sloanei seed, Global J. Pure Appl. Sci. 8 (1) (2002) 23–26.
- [22] F.U. Asoiro, S.L. Ezeoha, C.N. Anyanwu, N.N. Aneke, Physical properties of Irvingia gabonensis, Detarium microcapum, Mucuna pruriens and Brachystegia eurycoma seeds, Heliyon 6 (9) (2020) e04885, https://doi.org/10.1016/j.heliyon.2020. e04885.
- [23] M.M. Rahman, B.P. Lamsal, Ultrasound-assisted extraction and modification of plant-based proteins: impact on physicochemical, functional, and nutritional properties, Compr. Rev. Food Sci. Food Saf. 20 (2) (2021) 1457–1480.
- [24] M.N. Akhtar, Z. Mushtaq, N. Ahmad, M.K. Khan, M.H. Ahmad, A.I. Hussain, M. Imran, Optimal ultrasound-assisted process extraction, characterization, and functional product development from flaxseed meal derived polysaccharide gum, Processes 7 (4) (2019) e189, https://doi.org/10.3390/prf/040189.
- [25] J.F. Fabre, E. Lacroux, R. Valentin, Z. Mouloungui, Ultrasonication as a highly efficient method of flaxseed mucilage extraction, Ind. Crop. Prod. 65 (2015) 354–360, https://doi.org/10.1016/j.indcrop.2014.11.015.
- [26] I. Majid, G.A. Nayik, V. Nanda, Ultrasonication and food technology: a review, Cogent Food Agric. 1 (1) (2015) e1071022, https://doi.org/10.1080/ 23311932.2015.1071022.
- [27] A. Amiri-Rigi, S. Abbasi, M.N. Emmambux, Background, limitations, and future perspectives in food grade microemulsions and nanoemulsions, Food Rev. Int. (2022) 1–39, https://doi.org/10.1080/87559129.2022.2059808.
- [28] A. Amiri-Rigi, S.K. Pillai, M.N. Emmambux, Development of hemp seed oil nanoemulsions loaded with ascorbyl palmitate: effect of operational parameters, emulsifiers, and wall materials, Food Chem. 400 (2023) e134052, https://doi.org/ 10.1016/j.foodchem.2022.134052.
- [29] Other emerging gums: flaxseed gum, yellow mustard gum, and psyllium gums, in: J. Kang, Q. Guo, S.W. Cui, G.O. Edito, Phillips, P.A. Williams (Eds.), Handbook of Hydrocolloids, Woodhead Publishing, Cambridge, 2021, pp. 597–624, https://doi. org/10.1016/B978-0-12-820104-6.00030-9.
- [30] R. Hoover, F. Sosulski, Composition, structure, functionality, and chemical modification of legume starches: a review, Can. J. Physiol. Pharmacol. 69 (1) (1991) 79–92, https://doi.org/10.1139/y91-012.
- [31] S.R. Derkach, N.G. Voron'ko, Y.A. Kuchina, Intermolecular interactions in the formation of polysaccharide-gelatin complexes: a spectroscopic study, Polymers (Basel) 14 (2022) e2777, https://doi.org/10.3390/polym14142777.
- [32] D. Šeremet, K. Žižek, I. Žepić, M. Kovačević, L.N. Nodilo, D. Vrsaljko, Z. Katančić, K. Sokač, S. Kuzmić, D. Komes, Effect of guar gum-based carriers on the physical and bioactive properties of spray-dried delivery systems of ground ivy (Glechoma hederacea L.), Food Hydrocoll. 150 (2024) e109658, https://doi.org/10.1016/j. foodhyd.2023.109658.
- [33] E. Alpizar-Reyes, H. Carrillo-Navas, R. Gallardo-Rivera, V. Varela-Guerrero, J. Alvarez-Ramirez, C. Pérez-Alonso, Functional properties and physicochemical characteristics of tamarind (Tamarindus indica L.) seed mucilage powder as a

#### A.S. Adeboye et al.

novel hydrocolloid, J. Food Eng. 209 (2017) 68-75, https://doi.org/10.1016/j. jfoodeng.2017.04.021.

- [34] Z. Li, L. Jiang, Y. Wang, M. Li, T. Liu, Y. Liu, Chitosan–gellan gum polyelectrolyte hydrogel beads containing tea tree oil microcapsules: preparation, characterization and application, Food Hydrocoll. 157 (2024) e110464, https://doi.org/10.1016/j. foodhyd.2024.110464.
- [35] A.A. Attama, P.A. Akpa, Determination of the amorphicity and glass transition temperatures (Tg) of some natural polysaccharides, J. Drug Deliv. Technol. 18 (3) (2008) 219–220, https://doi.org/10.1016/S1773-2247(08)50040-2.
- [36] M.J. Zohuriaan, F.J.P.T. Shokrolahi, Thermal studies on natural and modified gums, Polym. Test. 2 (5) (2004) 575–579, https://doi.org/10.1016/j. polymertesting.2003.11.001.
- [37] L. Juszczak, M. Witczak, T. Ziêba, T. Fortuna, Rheological behaviour of heated potato starch dispersions, Int. Agrophys. 26 (4) (2012) 381–386, https://doi.org/ 10.2478/v10247-012-0053-3.
- [38] M. Marcotte, A.R.T. Hoshahili, H. Ramaswamy, Rheological properties of selected hydrocolloids as a function of concentration and temperature, Food Res. Int. 34 (8) (2001) 695–703, https://doi.org/10.1016/S0963-9969(01)00091-6.
- [39] M. Roullet, P.S. Clegg, W.J. Frith, Viscosity of protein-stabilised emulsions: contributions of components and development of a semi-predictive model, J. Rheol. 63 (2019) 179–190, https://doi.org/10.1122/1.5062837.
- [40] M.C.N. Picot-Allain, A. Amiri-Rigi, K. Abdoun-Ouallouche, L. Aberkane, A. Djefal-Kerrar, M.F. Mahomoodally, M.N. Emmambux, Assessing the bioactivity, cytotoxicity, and rheological properties of pectin recovered from citrus peels, Food Biosci. 46 (2022) e101550, https://doi.org/10.1016/j.fbio.2022.101550.
- [41] F. Sadeghi, R. Kadkhodaee, B. Emadzadeh, G.O. Phillips, Phase behavior, rheological characteristics and microstructure of sodium caseinate-Persian gum system, Carbohydr. Polym. 179 (2018) 71–78, https://doi.org/10.1016/j. carbpol.2017.09.060.
- [42] F. Sadeghi, R. Kadkhodaee, B. Emadzadeh, K. Nishinari, Effect of sucrose on phase and flow behavior of protein-polysaccharide mixtures, Food Hydrocoll. 113 (2021) e106455, https://doi.org/10.1016/j.foodhyd.2020.106455.
- [43] G. Stojkov, Z. Niyazov, F. Picchioni, R.K. Bose, Relationship between structure and rheology of hydrogels for various applications, Gels 7 (4) (2021) e225, https://doi. org/10.3390/gels7040255.
- [44] P. Matricardi, C. Cencetti, R. Ria, F. Alhaique, T. Coviello, Preparation and characterization of novel Gellan gum hydrogels suitable for modified drug release, Molecules 14 (9) (2009) 3376–3391, https://doi.org/10.3390/ molecules14093376.
- [45] T.T. George, A.B. Oyenihi, F. Rautenbach, A.O. Obilana, Characterization of Moringa oleifera leaf powder extract encapsulated in maltodextrin and/or gum Arabic coatings, Foods 10 (12) (2021) e3044, https://doi.org/10.3390/ foods10123044.
- [46] S. Fang, W. Qiu, J. Mei, J. Xie, Effect of sonication on the properties of flaxseed gum films incorporated with carvacrol, Int. J. Mol. Sci. 21 (5) (2020) e1637, https://doi.org/10.3390/ijms21051637.
- [47] C. Wu, D.J. McClements, L. Li, M. He, Y. Li, F. Teng, Fabrication of composite hydrogels by assembly of okara cellulose nanofibers and gum Arabic in ionic

liquids: structure and properties, J. Mol. Liq. 349 (2022) e118132, https://doi.org/ 10.1016/j.molliq.2021.118132.

[48] M. Salehi, M. Tabarsa, M. Amraie, M. Anvari, M. Rezaei, B.M. Smith, Characterization of rheological and structural properties of a gum from Balangu seeds, Int. J. Biol. Macromol. 117 (2018) 294–300, https://doi.org/10.1016/j. ijbiomac.2018.05.033.

# Glossary

- Mucuna sloanei: A legume species from which gum is extracted, known for its physicochemical properties and application as a thickener in food.
- Ultrasonication: A technique that uses high-frequency sound waves to enhance extraction efficiency and modify the properties of materials.
- Differential scanning calorimetry (DSC): A method for measuring thermal properties of materials, crucial for understanding gelatinisation and melting behaviours.
- Gelatinisation temperatures (To, Tp, Tc): Key temperatures indicating the onset, peak, and conclusion of the gelatinisation process, important for evaluating how material behaves in food applications.
- Thermogravimetric analysis (TGA): A technique that assesses thermal stability by tracking weight loss as temperature increases, indicating material decomposition.
- Degradation onset temperature (T5%): The temperature at which a 5 % weight loss is observed during TGA, serving as an indicator of thermal stability for materials.
- Rheological properties: Characteristics describing how materials flow and deform under applied stress, essential for understanding food texture and processing behaviour.
- Yield stress: The minimum stress required to initiate flow in a material, important for applications as a binder and to understand material cohesiveness.
- Power-law model (Ostwald-de Waele model): A mathematical model describing the flow behaviour of non-Newtonian fluids, characterised by parameters for consistency and flow behaviour.
- Zero shear viscosity ( $\eta_0$ ): The viscosity of a fluid at very low shear rates, reflecting its behaviour under quiescent conditions.
- Shear-thinning behaviour: A property of non-Newtonian fluids where the viscosity decreases with increasing shear rate, relevant for the processing and application of gums.
- Linear viscoelastic region (LVR): The range of applied stress or strain in which a material exhibits predictable linear viscoelastic responses.
- Storage modulus (G): A measure of the elastic behaviour of a material, indicating its ability to store energy during deformation.
- Loss modulus (G"): A measure of the viscous behaviour of a material, reflecting energy lost as heat during deformation.
- Residue percentage at Tmax and 600 °C: The remaining mass percentage of a material at its maximum degradation temperature and at 600 °C, providing insight into thermal decomposition patterns and structural integrity.
- Principal component analysis (PCA): A statistical method used to reduce data dimensionality and identify patterns, facilitating comparison among different samples.