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Preliminary formulation studies of water-soluble granules of *Hibiscus sabdariffa* calyx

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Abstract: **Background**: The formulation of *Hibiscus sabdariffa*, a plant reported to have hypoglycemic, antihypertensive, antibacterial, antioxidant and anti-inflammatory properties, into a suitable dosage form is vital in improving patient compliance and maximizing its benefits. **Objectives**: This study aims to formulate water-soluble granules from the calyx of *Hibiscus sabdariffa* using three (3) different granulating fluids and characterize the formulated granules to obtain an optimized formulation. **Methods**: Dried calyces of *Hibiscus sabdariffa* were pulverized and formulated into granules via the wet granulation technique using three different granulating fluids: xanthan gum mucilage (2 %), glucose solution (50 %), and syrup BP. Granule flow properties and solubility were determined. Interactions were monitored using Fourier transform infrared (FT-IR,), while morphological attributes were assessed using X-ray powder diffraction (XRD) and scanning electron microscopy. **Results**: The mean granule diameter of the granules was between 700 and 740 µm, and the moisture content was between 0.7 and 1.3 % with sucrose-based granules having the least amount of moisture. All batches had excellent flow. The insoluble particles and aqueous dispersibility were between 2.20 and 4.45 g and 158.67 and >2400 sec, respectively. Glucose-based granules were more soluble and easily dispersible. FT-IR showed no interaction between the granulating fluids and hibiscus calyces, while XRD of the optimized formulation (glucose-based granules) revealed decreased crystallinity. **Conclusion**: Glucose solution (50%) is considered the most suitable fluid for the formulation of water-soluble granules of *Hibiscus sabdariffa* calyces. This study demonstrates the potential of formulating an acceptable oral dosage form from the calyces of *Hibiscus sabdariffa* using a 50% glucose solution as a granulating fluid, yielding optimal granule properties that will enhance product acceptability and patient compliance.

Keywords: Characterization; Dispersibility; *Hibiscus sabdariffa; W*ater-soluble granules; Wet granulation

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INTRODUCTION

The use of herbal remedies for the treatment and management of diseases has its origins in ancient cultures but has now been embedded in the world's culture, including in developing countries, as an alternative medicine. The World Health Organization (WHO) reports that about 60% of the world's population depends on herbal medicine for health care and 80% of the population in developing climes depends almost totally on them as their source of primary health (Moh'd and Iqbal, 2019).

Many plant remedies have been reported to have the potential to treat both communicable and non-communicable diseases. The plant *Hibiscus sabdariffa* Linn, family Malvaceae, is also referred to as "Roselle." It is native to India and Malaysia but is widely grown in tropical and subtropical climates (Riaz and Chopra, 2018). In Nigeria, it is widely distributed in the Middle Belt States of Plateau, Nasarawa and Benue, as well as some South-Western states like Osun and Ogun (Aganbi *et al.*, 2017). The plant is popular because of its calyces, which are a high source of nutritional and medicinal benefits. Green calyces are consumed as vegetables by humans and animals and red calyces have been used in sauces and jams (Salami and Afolayan, 2020). The hot water extract of the red calyces is widely consumed as a refreshing drink or beverage and is commonly known as "Zobo drink" in Nigeria (Ojileh and Okechukwu, 2023). Traditionally, the extract of Roselle is useful as an antimicrobial, antispasmodic, diuretic, laxative, antidiabetic, cardioprotective, antihypertensive, nephroprotective, weight-reduction agent, among others (Da-Costa-Rocha *et al.*, 2014). Scientifically, these folklore claims have been investigated and corroborated to certain levels. Although literature reveals the great therapeutic potential of the calyces of Roselle, the lack of appropriate means of dosage presentation poses a challenge to modern-day users and limits its acceptability and adherence. Furthermore, the usual methods of using these herbs (decoction, infusion, etc.) impact the standardization and long-term stability of the bioactive components (Izah *et al.*, 2016).

An appropriate dosage form ensures that the drug efficiently gets to the desired part of the body. Granules are solid oral dosage forms consisting of dry aggregates of powder particles. The process of granulation is a common

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one in the food and pharmaceutical industries that turns small powders into bigger, discrete particles, or granules (Aulton and Summers, 2017). Several advantages of this approach include increased food and pharmaceutical production efficiency, functionality and overall quality (Aulton and Summers, 2017). Granulation reduces the bulk storage area required for the calyxes, making them easier to handle, transport and store than the whole unprocessed calyx. Processing into granules also makes them more suitable for usage in a variety of applications, it improves product standardization and shelf-life, providing a value-added product with wider market interests (Aulton and Summers, 2017; Parikh, 2021). This is crucial for business objectives as it guarantees a consistent product for customers (Swanborough, 2008). This study aims to formulate water-soluble granules from the calyx of *H. sabdariffa* using three (3) different granulating fluids and characterize the formulated granules to obtain an optimized formulation.

MATERIALS AND METHODS

Materials

Dried red roselle calyces purchased from Sheik Abubakar Gummi Market (Kaduna, Nigeria). Glucose (Evans Baroque, Nigeria), xanthan gum (Central Drug House, India), sucrose (Bua Sugar Refinery, Nigeria). Distilled water prepared in the Pharmaceutical Chemistry Laboratory, Kaduna State University.

Preparation of roselle calyces

The red roselle calyces were sorted to remove dirt and unwanted materials. They were identified and authenticated at the Department of Biological Sciences, Kaduna State University and assigned the voucher number; KASU/BSH/788. The samples were then dried at 40 °C in a hot air oven (model DHG-9030, USA) for 2 h.

Size-reduction of dried roselle calyces

The dried roselle calyces were pulverized into coarse powder using a tabletop laboratory blender (GEA TwinPANDA 400, Germany) and then milled into fine powder using a grain milling machine (GX LEON 120 machine, China). The powdered material was packaged in an air-tight container and kept in a desiccator until further analysis.

Particle size analysis

Fifty (50) grams of powdered calyces were transferred to a sieve shaker (Jin-Ling Shang, China). The sieves were set up as follows: pan collector, 0.15 mm, 0.18 mm, 0.28 mm, 0.45 mm and 0.9 mm, in ascending sequence. The sieve shaker was set to vibrate for 10 min, after which the particles retained on each sieve and collecting pan were weighed and recorded (Builder *et al.*, 2020). This evaluation was repeated three times.

Qualitative phytochemical screening

The powdered calyx was tested to determine the presence of alkaloids, saponins, flavonoids, glycosides, carbohydrates, tannins, anthraquinones and terpenoids using established methods (Evans, 2002; Harborne, 1998; Sofowara, 1993).

Granulation of powdered roselle calyx

The wet granulation technique of massing and screening was employed here. Three (3) granulating fluids, namely xanthan gum mucilage, syrup BP and glucose solution, were used for wet massing. Briefly, the process involved preparing xanthan gum mucilage (2 $\%^{w}/_{v}$) by dispersing 2g of xanthan gum in 100 mL of distilled water at room temperature and agitated for about 30 min to obtain a homogenous mixture. Powdered Roselle calyx (50 g) was gradually wet-massed with the mucilage until a wet mass formed and screened through a sieve with a 2 mm mesh size. The wet granules formed were dried in the hot air oven at 60 °C for 2 h. The dried granules were placed in a plastic bag with a silica gel sachet and stored for future evaluation.

Similarly, sucrose-based granules were prepared by wet-massing 50 g of roselle calyces with syrup BP that had been prepared according to the official method in British Pharmacopoeia (British Pharmacopoeia, 2015). The wet mass was screened, dried and packaged as done for the xanthan-based granules. Glucose-based granules were prepared by wet-massing 50 g of Roselle calyces with glucose solution (50 $\%^{w}/_{v}$) prepared by dispersing 50 g of glucose in 100 mL of distilled water and heating to 80°C on a hot plate while stirring until completely dissolved and a syrupy mixture was formed. Powdered roselle calyx (50 g) was gradually wet-massed with the mucilage until a wet mass formed. The wet mass was screened, dried and packaged as described above.

Granules moisture content determination

Exactly 3 g of each prepared granule was weighed into a crucible and dried in the hot air oven at 105 °C for 30 mins. Afterward, the crucibles were removed and transferred to a desiccator to cool for 5 minutes then taken out of the desiccator and each sample was weighed. The percentage moisture content was calculated using the formula in Equation 1 (Noma *et al.*, 2020):

$$\%\,\text{moisture content} = \frac{\text{Loss weight due to drying}}{\text{Weight of sample taken}} \times 100$$

Assessments of granule's aqueous dispersibility rate

The time taken for 5 g of the granules to be completely dispersed in 250 mL of water at room temperature (28 ± 2 °C) when stirred using a stirrer was noted. The measurements were taken in riplicate.

Assessments of insoluble matter

The method according to Dagogot *et al.*, (2020). was adopted. To estimate the amount of insoluble particles following aqueous dispersibility, 5 g of granules were weighed and placed into a conical flask containing 250 mL of distilled water (28 ± 2°C). The mixture was agitated for

40 mins on a rotary water bath and filtered through a Whatman filter paper. The residue was dried and weighed.

Granules flow rate determination

Thirty grams (30 g) of granules were loaded into the Erweka flow apparatus (Erweka G.M.B.H, Germany) and allowed to pass through the funnel orifice. The time (t) taken for the granules to pass through the aperture was recorded and the flow rate was calculated using the formula in Equation 2 (Dagogot *et al.*, 2020):

$$\text{Flow rate} = \frac{w}{t}$$

Angle of repose

The angle of repose was determined using the method described by Builders *et al.*, (2020). A funnel with an orifice of 1 cm was filled with 50 g of granules. The funnel was clamped to ensure its tip was 10 cm from the base of the flat surface. The granules were poured into the funnel whose tip had been. The orifice was opened and the granules were allowed to flow through to the flat surface. The height (H) and diameter (D) of the granule heap on the surface were measured and recorded. This was repeated three times. The angle of repose (ϴ) was calculated using Equation 3 (Mohammed *et al.*, 2022):

$$\text{Tan } \Theta^{O} = \frac{2H}{D}$$

Bulk density

Granules (25 g) were poured into a graduated cylinder (100 mL) which was left undisturbed on a flat surface. The bulk volume was determined by measuring the volume that the granules occupied. The bulk density was estimated using Equation 4(Mohammed *et al.*, 2022):

$$\text{Bulk Density} = \frac{\text{weight of granules (g)}}{\text{Bulk Volume (mL)}}$$

Tapped density

Granules (25 g) was poured into a graduated cylinder (100 mL) which was left undisturbed on a flat surface. The measuring cylinder was tapped unto a flat surface until constant volume was attained. The tapped density was determined using Equation 5 (Mohammed *et al.*, 2022):

$$\text{Tapped Density} = \frac{\text{Weight of granules (g)}}{\text{Tapped Volume (mL)}}$$

Hausner ratio (HR) and Carr's compressibility index (CI)

These were computed using data obtained from the bulk density (Db) and tapped density (Dt).

$$HR = \frac{Dt}{Db}$$

$$CI = \frac{Dt - Db}{Dt} X\ 100$$

Compatibility assessment

Fourier transform infrared (FT-IR) spectra of freshly prepared and 4-week-old granules were obtained to examine the formulation's characteristics (Mohammed *et al.*, 2022). The spectra were collected between 3000 and 1000 cm^{-1}.

Optimization criteria and further evaluation

Following a preliminary review of the three batches, the formulation with good organoleptic properties, fastest aqueous dispersibility rate and solubility was selected as the optimized batch and subjected to further evaluation.

Optimized batch stability assessment

Stability assessment of the optimized batch was carried out after 24 months of storage at ambient temperature (28 ± 2°C). Phytochemical profiling, mean granule size, flow property, solubility and dispersibility analysis and Fourier-transform infrared (FT-IR) spectroscopic analysis were done.

X-Ray diffraction

The X-ray diffraction patterns of powdered Roselle calyx and the optimized batch were obtained using a diffractometer. The scanning rate was 60 min^{-1} for the 10 to 500 diffraction angle (2θ) range.

Surface morphology

The surface morphology of the optimized batch was evaluated with a scanning electron microscope at magnifications of 500, 1000 and 2000. The sample was mounted on aluminum stubs and coated with a platinum-based electrical conducting substance applied with a vacuum coater.

Statistical analysis

Utilizing SPSS 20.0 software (SPSS, Chicago, IL, USA), the data collected were analyzed and provided as the mean ± standard deviation (SD). A *T*-test was used to compare data sets, with *p* -p-values < 0.05 indicating statistical significance.

RESULTS

Organoleptic and phytochemical properties of prepared granules

Fig. 1 shows that the dried calyces were observed to be burgundy in color, are brittle and have a coarse texture. It also shows that all the granules have the same burgundy color, are coarse and granular and have a coarse texture.

Phytochemical screening of the *H. sabdariffa* calyces revealed some bioactive substances, including carbohydrates, saponins, flavonoids and tannins as displayed in table 1.

Physicochemical and flow characteristics of prepared granules

Fig. 2 depicts a size-frequency distribution curve that was positively skewed. The highest point of the curve depicting the most common size range shifted to the left side of the x-axis, indicating that a greater proportion of granules fell into the smaller size range. The right side of the curve (the tail) gradually tapers off, suggesting that the number of granules decreases as size increases.

Table 1: Phytochemical constituents in *Hibiscus sabdariffa* calyces and granules

S/No.	Phytochemical group	*H. sabdariffa* calyx	*H. sabdariffa* granules Day 1	*H. sabdariffa* granules After 24 months
1.	Carbohydrate	+	+	+
2.	Saponins	+	+	+
3.	Flavonoid	+	+	+
4.	Terpenoids/steroid	-	-	-
5.	Glycoside	-	-	-
6.	Anthraquinones	-	-	-
7.	Alkaloid	-	-	-
8.	Tannins	+	+	+

Table 2: Physical properties of granules

Parameters		Formulations: Xanthan-based granules	Sucrose-based granules	Glucose-based granules (Optimal formulation) Day 1	Xanthan-based granules After 24 months
Mean granule diameter (μm)		740	700	714	714
Flow rate (g/s)		76±1.53[a]	149±21.54[a]	81.67±12.77	81.50±11.40
Angle of repose (^{O})		19.31±1.05[a]	13.81±1.3[a]	15.43±0.74	15.00±0.58
Hausner ratio		1.12	1.16	1.28	1.29
Carr's index		0.11	0.14	0.22	0.22
Amount of insoluble particles (g) (solubility assessment)		4.45±0.76[a]	2.33±0.59	2.20±0.31	2.13±0.83
Aqueous dispersibility rate (sec)		>2,400	164.67±15.06	158.67±16.69	155.32±9.93
Moisture content (%)	Day one granules	1.3	0.7	1.3	-
	4 months old granules	3.3	0.0	0.3	-
	24 months old granules	3.3	0.0	-	0.1

[a]Significant mean difference at a 5 % level (i.e. $p < 0.05$)

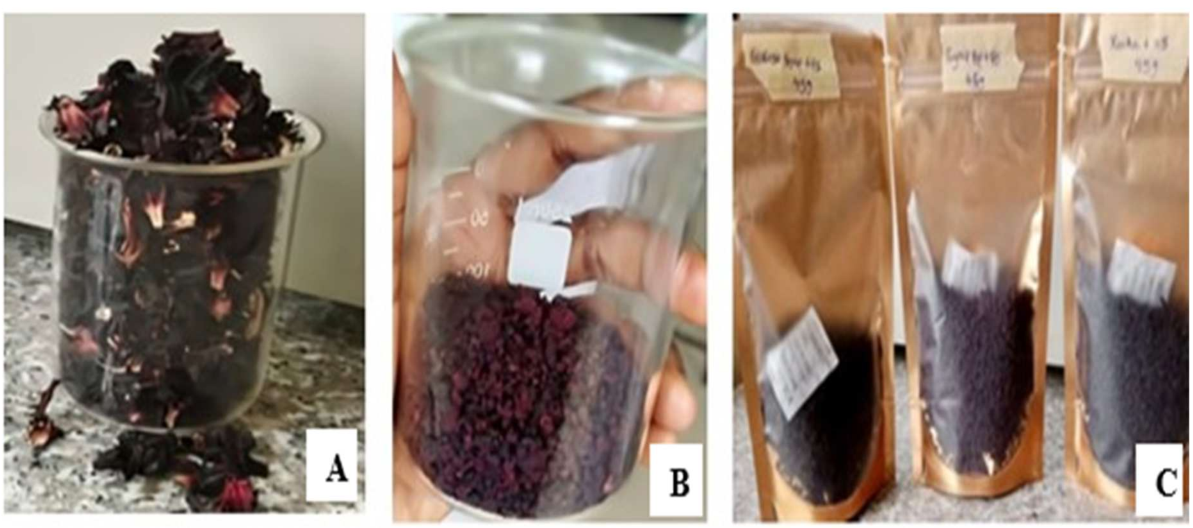

Fig. 1: Physical appearance of *Hibiscus sabdariffa* calyces at different stages: (A) dried calyces, (B) formulated granules, and (C) packed granules

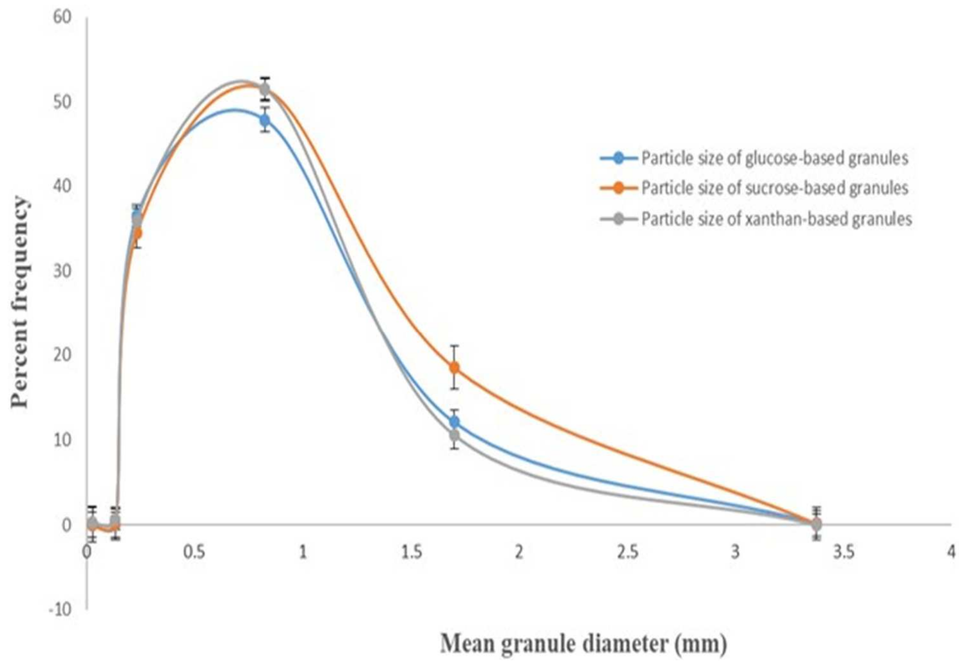

Fig. 2: Size-frequency distribution curves displaying the distribution of particle sizes and their frequencies for glucose, sucrose, and xanthan-based *Hibiscus sabdariffa* granules

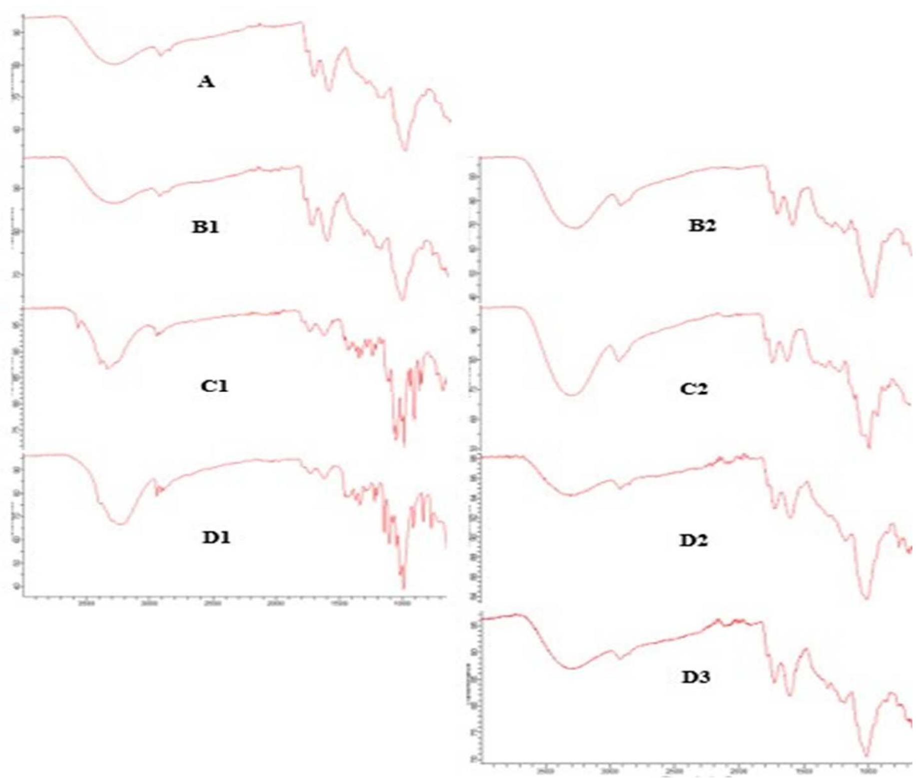

Fig. 3: FTIR spectra of samples A, B1, B2, C1, C2, D1, D2, and D3, illustrating the characteristic absorption bands for each sample to compare their functional groups and chemical compositions

A: Dry *Hibiscus sabdariffa* calyx B1: Freshly prepared xanthan-based *Hibiscus sabdariffa* granules B2: 4 weeks old xanthan-based *Hibiscus sabdariffa* granules C1: Freshly prepared sucrose-based *Hibiscus sabdariffa* granules C2: 4 weeks old sucrose-based *Hibiscus sabdariffa* granules D1: Freshly prepared glucose-based *Hibiscus sabdariffa* granules D2: 4 weeks old glucose-based *Hibiscus sabdariffa* granules D3: 24 months old glucose-based *Hibiscus sabdariffa* granules

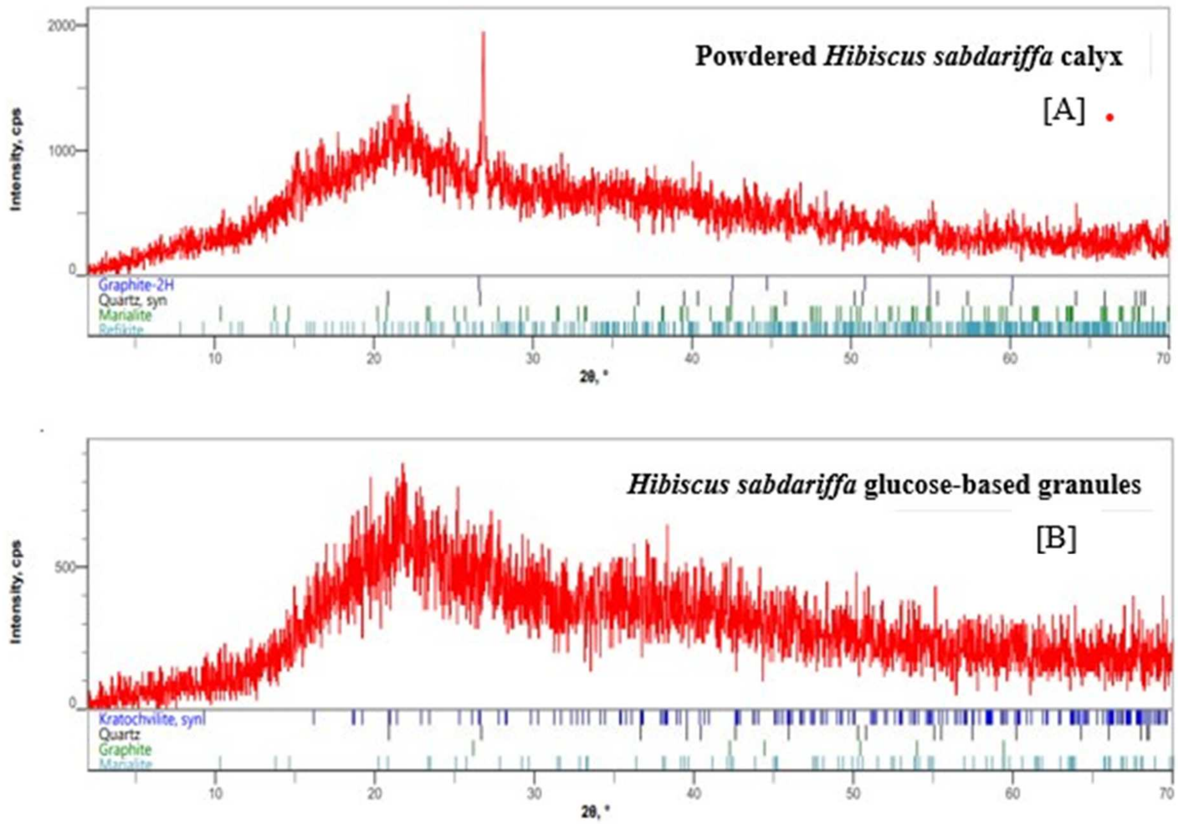

Fig. 4: X-ray diffraction (XRD) patterns comparing (A) powdered *Hibiscus sabdariffa* calyx with (B) granules made from *Hibiscus sabdariffa* using glucose as a binder

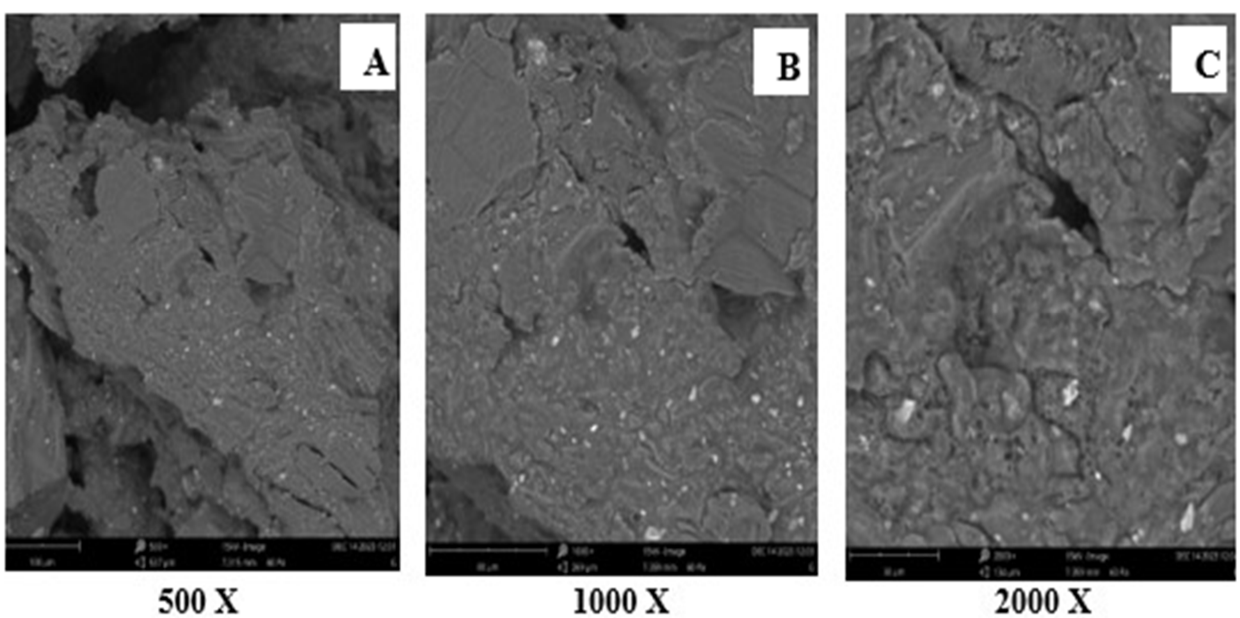

Fig. 5: Scanning electron micrograph of glucose-based *Hibiscus sabdariffa* granule illustrating its detailed morphology at different magnifications; (A) 500x, (B) 1000x and (C) 2000x

A lesser proportion of granules in the distribution that are larger than the most common sizes are represented by the right side.

Table 2 summarizes the physicochemical properties of all the formulated granules. It shows that all the granule formulations had a mean particle size of 700-740 µm, with xanthan-based granules having the largest size. All three formulations had angle of repose less than 20°, indicating excellent flow. The flow rate of the granules ranged from 76 g/sec to 149 g/sec, with sucrose-based granules having the highest flow rate. The bulk and tapped density values did not differ considerably across the formulations, ranging from 28.0-29.3 g/mL for bulk density and 22.0-25.0 g/mL for tapped density. The amount of insoluble particles following granule solubility assessment was in the following order: glucose-based granules < sucrose-based granules < xanthan-based granules, with glucose-based granules having the fastest solubility.

Glucose-based granules exhibited the fastest aqueous dispersibility rate (158.67 sec) in an aqueous medium at room temperature (28 ± 2 ºC), while xanthan-based granules showed the least aqueous dispersibility rate (>2,400 sec). The moisture content of freshly prepared granules was 1.3 % for xanthan-based granules, 0.7 % for sucrose-based granules and glucose-based granules was 1.3 %. After 4 months of storage, the percentage moisture content in sucrose-based granules and glucose-based granules was observed to decrease to 0 % respectively while that of xanthan-based granules was observed to have increased (3.3 %).

Fourier transform infrared spectroscopy, X-ray diffraction and surface morphology analysis of prepared granules

The FTIR spectrum of *H. sabdariffa* revealed a broad peak at 3276.3 cm^{-1}, indicating the presence of O-H stretching vibrations, likely from hydroxyl groups (alcohols, phenols and carboxylic acids) in compounds like polyphenols found in *H. sabdariffa* calyx. A medium sharp peak at 2922.2 cm^{-1} was detected, attributable to C-H stretching vibrations in organic compounds present in the calyx. Carbonyl groups (C=O stretching) are represented by a peak at 1733.2 cm^{-1}. A strong, distinct band (-C-O stretching vibration) is visible at 1013.8 cm^{-1}. After 4 weeks of storage, the FTIR spectra of xanthan-, sucrose- and glucose-based granules were similar to the spectrum of *H. sabdariffa* calyx as can be seen in fig. 3.

The X-ray diffraction pattern of the powdered *H. sabdariffa* calyces and the glucose-based *H. sabdariffa* granules are shown in fig. 4. The diffractogram of the pure powdered *H. sabdariffa* calyces showed several sharp and strong peaks within the range of 15-35 ϴº. The diffraction pattern of the glucose-based *H. sabdariffa* granule showed broadened diffraction peaks, which were less intense than that of the pure powdered *H. sabdariffa* calyces. The scanning electron micrograph of the glucose-based *H. sabdariffa* granules showed irregular shapes with a smooth surface texture, as can be seen in fig. 5.

DISCUSSION

Both pharmaceutical and food products/beverages must be elegant to provide a strong brand identity and foster positive associations with the product (Arab *et al*., 2022; Ilievska *et al*., 2016). The prepared granules of *H. sabdariffa* were found to be colorful, granular, distinct and visually appealing and have good organoleptic properties that are consistent with granule formulations.

Phytochemicals such as terpenoids, flavonoids, saponins, carbohydrates and tannins observed in this study are in tandem with those previously reported for *H. sabdariffa* calyces (Aliyu *et al*., 2014; Preciado-Saldaña *et al*., 2019). These biomolecules have been shown to have favorable effects on lipid metabolism, antioxidant, renal/diuretic, hepato- and nephroprotective (anti-cholesterol), antibacterial, antihypertensive and anti-diabetic properties, among others (Aliyu *et al*., 2014; Cid-Ortega and Guerrero-Beltrán, 2015; Da-Costa-Rocha *et al*., 2014).

Pharmaceutical formulations in granules provide greater stability compared with powders and are normally more palatable to patients where swallowing impairments exist, which is especially relevant in populations with dysphagia. On the other hand, tablets and capsules are associated with better compliance due to better convenience and effectiveness of the taste-masking processes. Granulates are however, useful in circumstances where flexibility in administration is crucial or where quick dissolution is considered important. The dose, dose frequency and organoleptic properties are, therefore, important in ensuring the optimal patient adherence and acceptability (Ahmed *et al*., 2024; Leyva-López *et al*., 2024; Nguyen *et al*., 2022). Granulating the calyx of *H. sabdariffa* yielded a more convenient product, potentially positioning it as a premium product that appeals to a larger variety of consumers.

An effective tool for understanding size variation within a population is a size-frequency distribution curve. It is useful in showing the distribution of sizes among a population of particles or granules (Aulton and Summers, 2017). The size-frequency distribution curve shows that the data obtained does not follow a normal distribution. A positively skewed curve (mode to the left of the mean) was obtained, indicating that more particles are smaller than the average size. This may be due to the presence of fragmented bits in the sample and could be related to the brittleness of the roselle. This underscores the importance of modifying processing conditions and optimizing the process to reduce fines formation. Methods such as granule screening, as well as the use of roller compaction or fluidized bed granulation, may be used to produce more uniform granules (Arndt *et al*., 2018; Parikh, 2021).

The flowability of powders or granules refers to how easily they move under the influence of an applied force or under the influence of their own weight (Aulton, 2017; Goh *et al.*, 2018). It is an essential characteristic in industries that handle and process bulk powders or granules. Powdered components, for example, must flow freely during food or pharmaceutical processing to be mixed, filled and packaged properly. Clumping or uneven flow can lead to manufacturing delays, waste and inconsistencies in the finished product (Aulton, 2017; Clayton, 2019). The sucrose-based granules exhibited the most favorable flow properties; their high flow rate, significantly ($p < 0.05$) higher than that of xanthan-based granules, indicates easy granular movement with minimal resistance and the low angle of repose suggests that the granules can form a steeper pile with better internal flow.

All three batches had relatively low angle of repose (< 20 °); however, the angle of repose for sucrose-based granules was observed to be lowest (13 °), suggesting they formed the steepest pile when poured and indicating excellent flow properties. Although the angle of repose of the sucrose-based granules was lower than that of the glucose-based granules, no significant difference ($p > 0.05$) was found between them. All three batches possessed excellent flow because their values were below 20° specified as standard for materials with excellent flow (Aulton, 2017; Clayton, 2019; Goh *et al*., 2018). Both xanthan-based granules and sucrose-based granules had Hausner ratio values less than 1.2 and Carr's indices below 0.15, which are generally considered indicative of excellent flowability (Clayton, 2019; Mohammed *et al*., 2022). Glucose-based granules had a slightly higher Hausner ratio (1.28) and Carr's index (0.22), suggesting fair flowability. Moisture content evaluation is crucial in pharmaceutical granules because it ensures the granules possess the appropriate physical properties, such as flowability and chemical stability. High moisture content might contribute to increased particle cohesion, poor flow and microbial growth in a product (Crouter and Briens, 2014; Lu *et al*., 2018). The moisture content of all three batches is relatively low (< 1.5 %). It is nonetheless noteworthy that flowability can be greatly impacted by even small amounts of moisture due to an increase in interparticle cohesion (Clayton, 2019). The sucrose-based granules (moisture content: 0.7%) had the lowest moisture content, potentially contributing to the better flow properties compared to the other batches. Xanthan-based granules and glucose-based granules had the same moisture content (1.3%) for both) which was higher than that of the sucrose-based granules. This high moisture content may hinder granule flowability to some extent. However, differences in moisture content were not substantial and other factors might play a more significant role. All three batches have a similar mean granule size (around 700 µm). Generally, larger granules tend to flow better than smaller granules (Aulton and Summers, 2017). However, granule size may not be responsible for the differences observed in the flow characteristics of the granules in this study. Overall, sucrose-based granules appear to have the best flow properties based on the highest flow rate and lowest angle of repose while xanthan-based granules and glucose-based granules showed comparable flow properties.

Aqueous dispersibility testing is important in determining the quality and usefulness of granular food/pharmaceutical products (Khader, 2016; Rowe *et al*., 2009). It evaluates how easily and uniformly granules disperse in aqueous medium. For a variety of reasons, granular food/pharmaceutical products are frequently desired to be easily dispersible in water. Dispersible products have fast dispersion rates, are fast and easy to prepare and this is convenient for consumers and improves their adherence and compliance with the product. They can simply add the granules to water and stir, avoiding the need for long periods of mixing or the concern of lumps forming. This characteristic is particularly important for products such as instant beverages. Easy dispersion makes the product more user-friendly. Consumers may produce meals or beverages fast, without the need for sophisticated mixing processes or long waiting periods. Quick dispersion is required for optimum dissolution and flavor release. This is especially true for products where the flavor must be immediately noticeable upon contact with water. Easy dispersion allows for quick and even incorporation into other foods or beverages. Granules that disperse well can be easily incorporated into other recipes, allowing for more versatile use of the product (Campbell-Platt, 2009; Khader, 2016). Based on the results of the dispersibility rate displayed in Table 2, the dispersibility ranking is as follows: glucose-based granules > sucrose-based granules > xanthan-based granules. The results show significant differences ($p < 0.05$) in aqueous dispersibility between xanthan and sucrose-based granules. The xanthan-based granules had the least aqueous dispersibility indicating that the granules may not disperse well in water even within a reasonable time, which defeats the aim of developing the product. Its high amount of insoluble particles (4.45 g) further supports this conclusion. Several factors could contribute to the poor dispersibility of the xanthan-based granules, such as the high molecular weight of the gum, which is capable of forming a tangled network in water thereby, hindering hydration. In addition, the strong hydrogen bonding within xanthan molecules can limit water penetration and slow down granule breakdown (Rowe *et al*., 2009). Glucose-based granules, on the other hand, had the fastest dispersion rate (< 3 min) indicating relatively quick and easy hydration. Consequently, the glucose-based granules had the least amount of insoluble particles (2.20 g) among the three batches.

The compatibility of extracts, active pharmaceutical ingredients (API) and excipients used in formulation manufacturing can be determined by FTIR spectrum analysis. If the API and excipient mixture (formulation) spectra have significant changes in distinctive peaks compared to the individual spectra, this could indicate interactions. The interaction has the potential to alter the end product's stability, efficacy and safety. When certain peaks in the formulation spectrum disappear in comparison to their components, it may indicate that the API is degrading because of an excipient incompatibility (Yahaya *et al*., 2017, 2024). No new peaks, shifting, or disappearance of peaks were observed in the FT-IR spectra of the three formulations compared to the spectrum of the *H. sabdariffa* calyx after 4 weeks of storage. This indicates a low likelihood of chemical interaction between the different components of the formulations, suggesting that the components are compatible. The phytochemical composition of *H. sabdariffa* calyces optimised into granules did not change during the storage period of 24 months. During the period, no significant difference ($p > 0.05$) was observed in the solubility, granule size, flow characteristics, or dispersibility. The FT-IR analysis of the optimised formulation revealed no significant variation in spectrum which indicates that it is physicochemically stable during the 24-month period.

Wet granulation is an effective way of granule production but may accelerate the degradation of certain bioactives in *H. sabdariffa* calyces-especially anthocyanins and ascorbic acid, by encouraging hydrolysis, oxidation and thermal degradation (M'be *et al.*, 2023). As a result, the variability of the formulation parameters, namely, the choice and amount of excipients (e.g., glucose), the pH gradient and control of the drying conditions, obtains special significance. These losses have been shown to be reduced by strategies like encapsulation, low temperature drying, optimum carrier concentrations and protective packaging both in granular and syrup formulations (Lema *et al*., 2022; M'be *et al*., 2023; Nguyen *et al*., 2022; Thamaraiselvi *et al*., 2025). An essential technique for evaluating formulations is X-ray diffraction, which provides important information about the physical form of a formulation (Goel *et al.*, 2022; Rostamabadi *et al.*, 2020). Peak broadening and reduction in intensity were observed in the diffraction peaks of the *H. sabdariffa* granule. This may be a result of a decrease in crystallite size, the presence of strain within the crystal lattice, or a decrease in crystallinity following the granulation of the sample (Dagogot *et al*., 2022). Further investigations are underway to elucidate the extent and impact of these structural changes.

The scanning electron microscopy (SEM) analysis provides valuable insights into the characteristics of the *H. sabdariffa* granules. The 2000x magnification provides more details when viewing the surface morphology of the granules. The observed irregular shapes in the SEM micrograph are a common characteristic of wet granulation processes (Clayton, 2019). Wet granulation involves agglomerating the powder particles using a binder solution, which can result in non-uniform granule shapes. The smooth surface texture suggests good uniformity of the granules with minimal surface defects like cracks or fissures. The granules' good flow characteristics are likely due to their smooth surface texture and satisfactory size range (700-740 µm). Good flow properties are essential for ensuring consistent and efficient dispensing of the granules during beverage production. Overall, the SEM analysis suggests that the wet granulation process was successful in producing *H. sabdariffa* granules with desirable characteristics. The granules have a satisfactory size range, irregular shapes typical of wet granulation, a smooth surface texture indicating acceptable homogeneity and excellent flow characteristics for ease of handling and dispensing.

CONCLUSION

The results of this study have shown that granulation of *H. sabdariffa* calyces with xanthan gum mucilage, syrup BP and glucose solution using the wet granulation technique can produce granule formulations with unique properties. However, granules produced with glucose solution exhibited rapid dispersibility and produced minimal insoluble matter upon dissolution. This study presents promising findings for the development of dispersible/ soluble granules from *H. sabdariffa* calyx and portrays the effect of the choice of granulating fluid on achieving specific granule properties for various applications.

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Authors' contributions
Zwanden Sule Yahaya and Danladi Dorcas Ayimiti conceived and designed the research. Zwanden Sule Yahaya, Boma Blessing Mohammed, and Olubunmi Jumoke Olayemi supervised the project and validated the results. Material preparation, investigation, and data analysis were performed by Danladi Dorcas Ayimiti, Oluwaseun Adenike Orugun, and Mponan Dung Guga. Zwanden Sule Yahaya and Olubunmi Jumoke Olayemi wrote the original draft. All authors reviewed and edited the draft, read and approved the final manuscript.

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Data availability statement
Data and material are available upon request.

Ethical approval
Not applicable

Conflict of interest

The authors declare no conflict of interest.

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