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Research Article

Formulation and development of capsules containing dry extracts from the calyces of *Hibiscus sabdariffa* L. and the sheaths of *Sorghum caudatum* H., intended as a dietary supplement

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Abstract

Introduction: Anthocyanin-rich extracts from *Hibiscus sabdariffa* L. calyces and *Sorghum caudatum* H. sheaths are promising antioxidants, but instability and dose variability limit use. This study aimed to standardize and manufacturing capsules containing lyophilized and purified dry extracts with affordable, plant-based excipients.

Methodology: An experimental program (Ouagadougou, Nov 2022–Aug 2023) prepared three extracts, lyophilized *H. sabdariffa*, purified *H. sabdariffa*, and purified *S. caudatum*. Physicochemical and pharmacotechnical tests assessed appearance, pH, hygroscopicity, residual moisture, and powder flow (angle of repose, Carr index, Hausner ratio). Total anthocyanins were quantified by differential pH (*H. sabdariffa*) and Stonestreet (*S. caudatum*). Size-0 capsules were formulated with maize starch and, when needed, colloidal silica; quality control evaluated appearance, mass uniformity (n=20), and disintegration (n=6).

Results: Extracts were acidic (pH 4.4–5.3), had residual moisture <10%, and were hygroscopic. Anthocyanins were 11.33±0.49 mg/g (lyophilized *H. sabdariffa*), 83.91±0.15 mg/g (purified *H. sabdariffa*), and 317.77±10.07 mg/g (purified *S. caudatum*). Flow was fairly good for lyophilized *Hibiscus* (Hausner 1.25) but poor to extremely poor for purified extracts (1.39–1.64); colloidal silica improved filling. Capsules were cylindrical, smooth, and compliant for mass uniformity; mean disintegration was 2.88–3.91 min.

Discussion: Capsules manufacturing enhanced standardized dose delivery, masked acidity, and limited environmental degradation. Gaps include granulometry, microbiological testing, post-formulation flow testing, and finished-product anthocyanin assay; ICH-aligned stability, dissolution in biorelevant media, and content uniformity are recommended.

Conclusion: Bench-scale standardization and capsules manufacturing yielded reproducible, rapidly disintegrating capsules suitable for supplementation and highlight the value of local botanicals.

Keywords: *Hibiscus sabdariffa* L., *Sorghum caudatum* H, anthocyanins, capsules

1. INTRODUCTION

Medicinal plants are a major source of bioactive substances used in therapy.^{1,2} Beyond their physiological roles in plants, defense against biotic and abiotic stressors and facilitation of reproduction, these

metabolites also exert relevant effects in the human body.³ Advances in organic chemistry during the second half of the twentieth century enabled the extraction and standardization of these compounds for medical and nutritional use.⁴ In industrialized countries, interest in phytomedicine has grown in response to modern

exposures (environmental pollutants, alcohol, medications, ultraviolet radiation, tobacco), which promote oxidative stress and metabolic disorders.^{5,6}

Oxidative stress is ambivalent: while reactive species are essential for cellular signaling, detoxification, and host defense, their excess contributes to the onset or progression of many diseases (cancer, cataract, pulmonary edema, accelerated aging).⁷ Preventing redox imbalance with antioxidants—substances capable of preventing or slowing the oxidation of other molecules—has therefore become a public-health and research priority.^{8,9} Numerous plant extracts exhibit documented free-radical-scavenging activity,¹⁰ notably the calyces of *Hibiscus sabdariffa* Lemordant, D. and the sheaths of *Sorghum caudatum* (Hack.) Stapf, valued for their richness in anthocyanins and other polyphenols with potential roles in preventing oxidative-stress-associated pathologies.¹¹⁻¹³ Accordingly, antioxidants aim to reduce the risk of various diseases¹⁴ and represent a scientific and societal priority by reinforcing endogenous defenses against oxidative stress.¹⁵

However, anthocyanins, water-soluble pigments ranging from red to blue, often show limited bioavailability and instability toward pH, temperature, and light, which may attenuate *in vivo* effects.¹⁶⁻²⁰ Dietary intakes vary widely and do not, by themselves, ensure optimal exposure.^{21,22} Moreover, quantitative extraction and purification are challenging (influenced by pH, solvent, temperature, and solid-to-solvent ratio) and must rely on food/industry-compatible processes (acidified ethanol and/or water).^{23,24} In this context, pharmaceutical formulation of standardized oral solid forms can improve stability, dose reproducibility, and clinical acceptability of anthocyanin-rich extracts.

Preliminary work at the Laboratory of Organic Chemistry and Applied Physics, Joseph KI-ZERBO University (Burkina Faso), has established extraction and quantification of total anthocyanins, total polyphenols, and antioxidant activity from *H. sabdariffa* and *S. caudatum* extracts.^{25,26} In line with regulatory definitions, food supplements are foods intended to supplement the normal diet and constitute concentrated sources of nutrients or other substances with nutritional or physiological effects, alone or in combination.²⁷ Developing capsules containing purified dry extracts of locally available antioxidant-rich plants thus aligns with a strategy of nutritional prevention under quality-assured conditions.

The study tests the hypothesis that standardizing and encapsulating purified dry extracts from the calyces of *Hibiscus sabdariffa* and the sheaths of *Sorghum caudatum* will produce stable, dose-reproducible oral dosage forms suitable for dietary supplementation, while preserving the antioxidant functionality of their anthocyanins. In line with this hypothesis, the work seeks to advance capsule-based food supplements from these extracts by formulating optimized capsules for each plant, manufacturing pilot experimental batches, and performing comprehensive quality control on both the extracts and the finished capsules, covering compliance, quantitative assay, and technological performance, to

maximize anthocyanin stability and ensure accurate dosing.

2. MATERIALS AND METHODS

2.1. Study design and setting

The experimental study was carried out in Ouagadougou, Burkina Faso, from November 2022 to August 2023. Extraction, characterization, and analytical assays were performed at the Laboratory of Organic Chemistry and Applied Physics (LCOPA, UFR/SEA). Powder and capsule quality control were conducted at the Galenics Laboratory of the Department of Traditional Medicine–Pharmacopoeia and Pharmacy (MEPHATRA/PH) at IRSS. Lyophilization, capsule preparation, and additional quality control took place at the Drug Development Laboratory (LADME) of Joseph KI-ZERBO University. Milling of the plant material was undertaken at the Department of Natural Substances of IRSAT.

2.2. Materials

➤ Plant materials and sample preparation

Calyces of *Hibiscus sabdariffa* Lemordant, D. (bissap) were purchased as mixed-lot (“tout-venant”) samples from Ouagadougou markets (August 2021, March 2022). Dry sheaths of *Sorghum caudatum* (Hack.) Stapf (red sorghum) were available at LCOPA. Calyces and sheaths were milled to powder using an electric grinder at IRSAT and stored desiccated until use.

➤ Equipment, glassware, and consumables

Balances: analytical (max 160 g, d = 0.1 mg; METTLER TOLEDO, Switzerland) and precision (max 1500 g, d = 0.1 g; METTLER, Switzerland); freeze-dryer (Alpha 1-4 LSC basic, CHRIST, Germany); rotary evaporator (BUCHI, Heating Bath B-300); disintegration tester (SOTAX DT2); pH meter (WTW 3210 SET3); halogen moisture analyzer (RADWAG); hotplate (DIAB MS-H280 Pro); powder flow tester (diameter/angle). Capsule shells (size 0), semi-automatic capsule filler, and opaque plastic bottles were used for manufacturing and packaging. Standard laboratory glassware (graduated cylinders, beakers, mortars/pestles, spatulas, filter papers) were employed.

➤ Reagents, excipients, and solvents

Colloidal silica (Aerosil® 200, Fagron, Saint-Denis); maize starch (diluent); aluminum chloride (LABKEM, 25 °C); standards: Trolox, quercetin, tannic acid; solvents: distilled water, absolute ethanol, absolute methanol; Amberlite XAD-7 resin; trifluoroacetic acid (TFA). Buffers for spectrophotometry are specified below.

2.3. Methods

➤ Extraction Methods and Dry Extract Characterization

- Extraction Procedures

Plant powders were processed to yield three extract types: a lyophilized aqueous extract from *Hibiscus sabdariffa* calyces, a purified phenolic-rich extract from the same calyces, and a purified phenolic-rich extract from *Sorghum caudatum* sheaths. Each extract then served as the active ingredient for its corresponding capsule formulation.

- *Hibiscus sabdariffa* — *Lyophilized Aqueous Extract*

One hundred grams of calyx powder were macerated in 300 mL of water for 24 h at 4 °C. The aqueous extract was centrifuged and filtered through paper; the process was repeated three times. Combined filtrates were lyophilized. The lyophilizate was stored at 4 °C in sterile bottles pending qualitative and quantitative analyses.

- *Hibiscus sabdariffa* — *Purified Phenolic-Rich Extract*

A second batch of 100 g of calyx powder was macerated in 300 mL of water; after centrifugation, the filtrate was percolated over an Amberlite XAD-7 column. Phenolics were adsorbed onto the resin, while mineral salts, sugars, and free organic acids were removed by extensive water washing. Phenolic compounds were eluted with ethanol acidified with 1% TFA to afford a phenolic-rich aqueous fraction, which was concentrated under reduced pressure (< 40 °C) to near dryness using a BUCHI rotary evaporator. The concentrate was redissolved in ethanol and precipitated in neat ethyl acetate. The precipitate was vacuum-dried and stored at 4 °C for analyses.

- *Sorghum caudatum* — *Purified Phenolic-Rich Extract*

One hundred grams of sheath powder were macerated for 24 h in 400 mL of acidified ethanol (ethanol:TFA 99:1, v/v). The extract was centrifuged and filtered; the process was repeated three times. Combined filtrates were concentrated under vacuum to near dryness, taken up in 15 mL ethanol, and precipitated into acidified distilled water. The precipitate was vacuum-dried and stored at 4 °C for analyses.

- *Extraction Yield*

Extraction yield (R, %) was calculated as the mass of dry extract obtained divided by the initial 100 g of plant powder, multiplied by 100: $R (\%) = (\text{mass of dry extract} / 100 \text{ g}) \times 100$.

➤ *Characterization of Dry Extracts*

- *Macroscopic and Organoleptic Features*

Color, taste, odor, and general appearance were recorded by direct observation.

- *Powder Flow Properties*

Angle of repose (α) was determined by funnel method (standard orifice), recording cone height and diameter after discharge. Compressibility (Carr index) and Hausner ratio were computed from untapped volume (V_0) and tapped volume (V_f) measured in a graduated cylinder: Carr index (%) = $100 \times (V_0 - V_f) / V_0$; Hausner ratio = V_0 / V_f . Results were interpreted against European Pharmacopoeia flowability scales.

- *Hygroscopicity*

Approximately 1 g of previously dried powder was placed in a desiccator over a saturated ammonium chloride solution at 25 °C for 24 h. Mass gain (%) was calculated as $100 \times (m_3 - m_2) / (m_2 - m_1)$, where m_1 = mass of empty container, m_2 = mass of container +

powder at T_0 , and m_3 = mass at 24 h. Classification: deliquescent (solution forms), very hygroscopic ($\geq 15\%$), hygroscopic ($\geq 2\%$ and $< 15\%$), slightly hygroscopic ($\geq 0.2\%$ and $< 2\%$).

- *Residual Moisture (Loss on Drying)*

About 1 g of each dry extract was weighed in triplicate watch glasses and dried in an oven at 105 ± 2 °C for 90 min to constant weight. Residual moisture (%) was computed as $100 \times (M_1 - M_2) / M$, where M_1 = mass before drying (glass + sample), M_2 = mass after drying (cooled), and M = initial sample mass.

- *pH Determination*

A 1% (w/v) dispersion of each purified or lyophilized extract was prepared in distilled water at 37 °C. After homogenization, pH was measured in triplicate using a calibrated pH meter, rinsing the electrode with distilled water between readings. Mean \pm SD values were reported.

➤ *Determination of Total Anthocyanins*

- *Hibiscus sabdariffa* — *Differential pH Method*

Total monomeric anthocyanins were estimated using a SAFAS MP96 microplate spectrophotometer via the differential pH method with two buffers: KCl, pH 1.0 (0.025 M) and acetate, pH 4.5 (0.4 M). For each sample, 30 μ L extract were mixed with 210 μ L buffer; absorbances were read after 15 min at 510 and 700 nm against a reagent blank. $A = (A_{510} - A_{700})_{\text{pH 1.0}} - (A_{510} - A_{700})_{\text{pH 4.5}}$. Anthocyanins (mg/L, as cyanidin-3-glucoside) were calculated by Beer-Lambert: $[\text{Anthocyanins}] = (A \times M \times D \times 1000) / (\epsilon \times l)$, with $M = 449.2 \text{ g}\cdot\text{mol}^{-1}$, $\epsilon = 26900 \text{ L}\cdot\text{mol}^{-1}\cdot\text{cm}^{-1}$, D = dilution factor, and l = path length (cm).

- *Sorghum caudatum* — *Stonestreet Method*

Total anthocyanins were quantified using a SAFAS DES 190 dual-energy spectrophotometer according to the Stonestreet method, employing buffers at pH 0.6 (KCl, 0.025 M) and pH 3.5 (acetate, 0.4 M). Samples were diluted (1:9, v/v) in each buffer, and absorbance was read after ~30 min at λ_{max} (apigeninidin ≈ 480 nm; path length = 1 cm). Based on $A = \epsilon \cdot l \cdot c$, concentration was derived from $\Delta A / \Delta \epsilon \cdot l$. Using the established conversion, $C (\text{mg/mL}) = \Delta A \times 661 \times 10^{-3}$, then converted to mg/L and expressed as mg per g of dry extract.

➤ *Capsule Formulation and Manufacturing*

- *Formulation Principle*

Capsules were designed to disintegrate rapidly and release the active extract, using powder blends with suitable flowability.

- *Extract Quantities Used*

Quantities obtained after extraction and amounts remaining after physicochemical tests are shown in Table 3.

Extract	Quantity obtained after extraction (mg)	Quantity available after tests (mg)
Lyophilized dry extract of <i>H. sabdariffa</i> calyces	12990	9006.2
Purified dry extract of <i>H. sabdariffa</i> calyces	5810	847.120
Purified dry extract of <i>S. caudatum</i> sheaths	10440	295.2

- Capsule Size Determination and Process Description

Capsule size was determined from the ratio of powder volume to the planned number of capsules (size \approx volume per capsule). Although calculations indicated sizes 1–2 for some batches, only size 0 hard gelatin capsules (nominal capacity 0.68 mL) and the corresponding semi-automatic filler were available at LADME. Therefore, batch sizes and excipient quantities were adjusted to fill size 0 capsules appropriately.

- Manufacturing Procedure

Powder volumes of extracts and excipients were measured, then blended by geometric dilution in a mortar. Blends were filled into size 0 capsules using a semi-automatic capsule filler. Empty capsules were separated (body/cap), bodies were filled, and capsules were closed with matching caps. This procedure was applied to all three extract types.

- Excipients

Colloidal silica (Aerosil 200) served as a glidant to improve blend flow and prevent agglomeration. Maize starch acted as an inert diluent/filler to reach the required capsule volume and facilitate homogeneous mixing.

- Dosage Estimations and Final Formulations

A target daily anthocyanin intake of 2.5 mg was specified. Regarding the *Hibiscus sabdariffa* lyophilized extract (anthocyanins 11.3 mg/g, *i.e.*, 1.13%), the required extract mass was 221.23 mg/day (10 days: 2212.3 mg). From the inferred bulk density (\approx 428.86 mg/mL; 9006.2 mg \approx 21 mL), the volume per dose was \sim 0.515 mL, enabling administration as one size-0 capsule (0.68 mL) per day; producing 40 capsules required a total fill of 27.2 mL, thus \sim 6.6 mL of maize starch was added as diluent.

For the purified *H. sabdariffa* extract (83.91 mg/g; 8.39%), the daily extract requirement was 30.12 mg (10 days: 301.20 mg); with an inferred bulk density of \sim 84.12 mg/mL (847.12 mg \approx 10 mL), the per-dose volume was \sim 0.351 mL, also suitable for one size-0

capsule per day; manufacturing 20 capsules (total 13.6 mL) required \sim 6.58 mL of excipient consisting of Aerosil 200 (0.69%) plus maize starch.

Concerning the purified *Sorghum caudatum* extract (317.77 mg/g; 31.77%), the daily extract need was 7.88 mg (10 days: 78.8 mg); using an inferred bulk density of \sim 26.84 mg/mL (295.2 mg \approx 11 mL), the per-dose volume was \sim 0.30 mL, again compatible with one size-0 capsule per day; producing 30 capsules (20.4 mL) required \sim 11.52 mL of excipient with Aerosil 200 at 0.37% plus maize starch.

The resulting batch compositions were as follows: *H. sabdariffa* lyophilized—8983.65 mg extract and 3405.1 mg maize starch, *q.s.* to 40 size-0 capsules (1 cap/day); purified *H. sabdariffa*, 694.67 mg extract, 7.01 mg Aerosil 200, and 305.69 mg maize starch, *q.s.* to 20 size-0 capsules (1 cap/day); purified *S. caudatum*, 266.63 mg extract, 2.98 mg Aerosil 200, and 544.4 mg maize starch, *q.s.* to 30 size-0 capsules (1 cap/day).

➤ Packaging and Labelling

Filled capsules were packaged in opaque white plastic bottles and labeled with: laboratory name, preparation name, composition, manufacturing date, expiry date, and batch number.

- Quality Control of Capsules

Quality control was performed according to European Pharmacopoeia (6th ed.) requirements, including macroscopic inspection, uniformity of mass, and disintegration testing.

- Macroscopic Characteristics

Appearance, color, and sealing integrity of capsules were recorded.

- Uniformity of Mass

Twenty capsules were randomly selected and weighed individually (filled), then emptied and the shells weighed. Net fill mass per capsule was computed by difference; the mean mass was compared with pharmacopeial limits (Table 4).

Dosage Form	Mean mass [mg]	Permitted deviation [% of mean]
Capsules, uncoated granules and powders (unit dose)	< 300 mg	\pm 10% (max 2 units outside; none beyond limits)
	\geq 300 mg	\pm 7.5% (max 2 units outside; none beyond limits)

- Disintegration Time

Six capsules were tested in water at 37°C using a standard disintegration apparatus (mesh-bottom tubes with optional disk). Disintegration was defined as the absence of palpable core, with only soft residue or envelope fragments remaining. Compliance was verified against the specified time limit per Ph. Eur.

3. RESULTS

3.1. Physicochemical Characterization of Dry Extracts

➤ Macroscopic and Organoleptic Properties

Macroscopic and organoleptic characteristics of the lyophilized and purified extracts of *Hibiscus sabdariffa* and the purified extract of *Sorghum caudatum*, Stapf are

Extract	Angle of repose (°)	Carr's index (%)	Hausner ratio	Flowability
Lyophilized <i>H. sabdariffa</i>	37.5	20.00	1.25	Fairly good
Purified <i>H. sabdariffa</i>	66.5	39.13	1.64	Extremely poor
Purified <i>S. caudatum</i> Stapf	48.5	28.20	1.39	Poor

➤ Hygroscopicity, Residual Moisture (Loss on Drying) and pH

All extracts were classified as hygroscopic (mass gain \geq 2% and $<$ 15%). Mean \pm SD values were 5.88 \pm 1.10% for lyophilized *H. sabdariffa*, 7.45 \pm 1.80% for purified *H. sabdariffa*, and 3.51 \pm 0.06% for purified *S. caudatum*.

Residual moisture contents were 6.00 \pm 0.03% for lyophilized *H. sabdariffa*, 5.60 \pm 0.01% for purified *H. sabdariffa*, and 8.28 \pm 0.05% for purified *S. caudatum*, all below the 10% specification.

Measured pH values were 4.4 (lyophilized *H. sabdariffa*), 4.9 (purified *H. sabdariffa*), and 5.3 (purified *S. caudatum*), indicating acidic profiles for all extracts.

➤ Total Anthocyanin Content

Total anthocyanins were lowest in the lyophilized *H. sabdariffa* extract (11.33 \pm 0.49 mg/g), higher in the

summarized below. The lyophilized *H. sabdariffa* extract appeared dark red, with a characteristic odor and a very sour taste, presenting a porous aspect. The purified *H. sabdariffa* extract was bright red with a characteristic odor, a sour taste, and a fine powder aspect. The purified *S. caudatum* extract was red with weakly characteristic odor and taste and a moderately fine powder aspect.

➤ Powder Flowability

Flow indices derived from angle of repose, Carr's compressibility index, and Hausner ratio indicated distinct behaviors across extracts. The lyophilized *H. sabdariffa* extract exhibited fairly good flow, whereas the purified *H. sabdariffa* and purified *S. caudatum* extracts showed extremely poor and poor flow, respectively.

purified *H. sabdariffa* extract (83.91 \pm 0.15 mg/g), and highest in the *S. caudatum* extract (317.77 \pm 10.07 mg/g). The purified *H. sabdariffa* extract contained approximately eightfold more anthocyanins than the lyophilized counterpart.

➤ Extraction Yield

From 100 g of plant powder, yields were 22.6% for lyophilized *H. sabdariffa*, 8.1% for purified *H. sabdariffa*, and 6.3% for purified *S. caudatum*.

3.2. Capsule Formulation, Manufacturing, and Packaging

➤ Formulations and Manufacturing

Formulations combined pharmaceutical excipients with active extracts, accounting for anthocyanin content determined previously. Batch compositions and pack sizes are reported below.

Extract	Active (mg)	Aerosil 200 (mg)	Maize starch (mg)	Pack size
<i>H. sabdariffa</i> lyophilized	8983.00	-	3405.10	40 caps, size 0
<i>H. sabdariffa</i> purified	694.67	7.01	305.69	20 caps, size 0
<i>S. caudatum</i> purified	266.789	2.98	544.40	30 caps, size 0

➤ Packaging and Labelling

Capsules were packaged in opaque white secondary boxes with corresponding labels. Label codes were: BL (bissap, lyophilized), BP (bissap, purified), and SP (sorghum, purified).

3.3. Quality Control of Capsules

➤ Macroscopic Characteristics

All capsules were cylindrical, smooth, clean, and properly sealed. Color was dark red for lyophilized *H. sabdariffa*, bright red for purified *H. sabdariffa*, and red for purified *S. caudatum*.

➤ **Uniformity of Mass**

Individual capsule masses met pharmacopeial criteria. Mean (\pm SD) net fill masses ($n = 20$) were 0.4344 ± 0.0093 g for lyophilized *H. sabdariffa*, 0.2259 ± 0.0064 g for purified *H. sabdariffa*, and 0.2755 ± 0.0108 g for purified *S. caudatum*; all were compliant.

➤ **Disintegration**

All formulations disintegrated within the 15-minute limit. Mean (\pm SD) disintegration times ($n = 6$) were 3.60 ± 0.41 min for lyophilized *H. sabdariffa*, 3.91 ± 0.13 min for purified *H. sabdariffa*, and 2.88 ± 0.26 min for purified *S. caudatum*; the purified *S. caudatum* capsules disintegrated fastest.

4. DISCUSSION

4.1. Study Limitations

This work aimed to develop capsule formulations using dry extracts from the calyces of *Hibiscus sabdariffa* L. and the sheaths of *Sorghum caudatum* H. as dietary supplements, while characterizing key physicochemical parameters. Several limitations should be noted. First, granulometric analysis of the plant powders prior to extraction, required to confirm particle-size distributions, was not performed because insufficient material was available. Second, microbiological testing of either the bulk extracts or finished capsules was not conducted. Third, post-formulation flow testing of the new blends was not undertaken, and the total anthocyanin content of the finished powders/capsules was not assayed to verify label claim. These gaps should be addressed in subsequent work to strengthen manufacturability and quality assurance.

4.2. Physicochemical Characterization of Dry Extracts

➤ **Macroscopic and Organoleptic Attributes**

Visual, tactile, and gustatory inspection differentiated the three extracts. The lyophilized *H. sabdariffa* extract was porous, dark red, and characteristically fragrant with a very sour taste; the purified *H. sabdariffa* extract was bright red, fine, and similarly characteristic in odor with a sour taste; the purified *S. caudatum* extract was moderately fine, red, with weakly characteristic odor and no specific taste. These findings indicate partial preservation of plant-specific sensory signatures. The chosen extraction approaches,²⁵ notably lyophilization, appear to have contributed positively to organoleptic retention despite higher cost, which is consistent with the recognized ability of freeze-drying to better preserve color, odor, and taste.

➤ **Acidity (pH)**

Aqueous pH values were acidic across extracts (≈ 4.4 for lyophilized *H. sabdariffa*; ≈ 4.9 for purified *H. sabdariffa*; ≈ 5.3 for purified *S. caudatum*). Such acidity is broadly compatible with gastric pH and may mitigate irritation while favoring rapid gastric dissolution and subsequent absorption, noting that pH is a determinant of gastrointestinal drug absorption.²⁸

➤ **Residual Moisture and Hygroscopicity**

Residual moisture contents were below 10% for all extracts ($\approx 6.0\%$ lyophilized *H. sabdariffa*; $\approx 5.6\%$ purified *H. sabdariffa*; $\approx 8.3\%$ purified *S. caudatum*), complying with European Pharmacopoeia (Ph. Eur.) and Codex limits for dried herbal preparations.²⁹ Lower water content typically confers improved microbiological stability³⁰ and reduces the likelihood of moisture-driven enzymatic reactions.³¹ Nevertheless, all extracts were classified as hygroscopic (mass gain $\geq 2\%$ and $< 15\%$ per Ph. Eur. 6th ed.),³² implying a propensity to absorb ambient humidity during handling and storage. To prevent flow deterioration via agglomeration and ensure dose uniformity, production should occur in humidity-controlled areas, with storage in airtight containers and, where appropriate, inclusion of desiccants.³³

➤ **Powder Flow and Rheology**

Flow indices (angle of repose, Carr's index, and Hausner ratio) distinguished the three extracts: the lyophilized *H. sabdariffa* showed fairly good flow (Hausner ≈ 1.25), whereas the purified *H. sabdariffa* and purified *S. caudatum* exhibited extremely poor and poor flow, respectively (Hausner ≈ 1.64 and ≈ 1.39). Particle size and distribution, shape, and density are known to govern flow and dosing behavior.³⁴ In this study, addition of a glidant (Aerosil 200) was necessary to correct poor flow and support reliable capsule filling, thereby improving the prospects for mass uniformity and dose reproducibility.³⁵

4.3. Capsule Characteristics and Performance

All capsules displayed acceptable macroscopic quality, cylindrical, smooth, clean, and non-deformed, indicating appropriate equipment condition and satisfactory process control with the semi-automatic filler. Capsule mass uniformity complied with Ph. Eur. acceptance criteria, supporting consistency of unit doses across batches. Disintegration occurred well within the 15-minute limit, with the purified *S. caudatum* capsules disintegrating fastest. Given that the shells were gelatin, rapid disintegration is expected and suggests prompt release in the gastrointestinal tract without capsule-derived toxicity.

4.4. Formulation Considerations

Formulations combined dry plant extracts as actives with maize starch (diluent/disintegrant) and Aerosil 200 (glidant). Both excipients are widely used in oral solid dosage forms and have no notable adverse effects at the proportions employed.³⁶⁻³⁸ Maize starch supports bulk-up to target fill volumes and facilitates disintegration, while colloidal silica improves blend flow by decreasing interparticle cohesion and adsorbing surface moisture.³⁷⁻⁴⁰ Capsules manufacturing offers multiple advantages in this context: masking the acidic taste of the extracts to enhance acceptability; improving dose reproducibility; and protecting anthocyanins, sensitive to heat, oxygen, light, and metal catalysts, from environmental degradation. The use of opaque, oxygen-impermeable secondary packaging further limits oxidative color changes and preserves activity.

4.5. Implications and Future Work

Future work will address the current gaps by conducting particle-size analysis (granulometry) of both source powders and finished blends, introducing microbiological testing for bulk extracts and final dosage forms, characterizing post-formulation flow properties, and assaying total anthocyanins in completed capsules to substantiate the label claim. In parallel, stability studies, accelerated and long-term in accordance with ICH guidance, together with water-activity control, dissolution profiling in biorelevant pH media, and content-uniformity testing will be implemented to reinforce quality, manufacturability, and shelf-life projections.

From a formulation standpoint, exploration of alternative fillers (*e.g.*, microcrystalline cellulose) or flow modifiers, optimization of capsule size and fill-weight, and evaluation of desiccant systems may further enhance manufacturability and patient acceptability, while maintaining the intended daily anthocyanin dose.

5. CONCLUSION

The primary objective of this study was to develop a solid dosage form using dry extracts from the lyophilized and purified calyces of *Hibiscus sabdariffa* L. and the purified dry extract from the sheaths of *Sorghum caudatum* H., formulated with cost-effective, naturally derived excipients. In parallel phytochemical work, anthocyanins were identified as the principal chemical group of interest.

We proposed and produced a capsule dosage form based on these extracts and evaluated the pharmaco-technical characteristics of the three anthocyanin-rich materials. The lyophilized and purified *H. sabdariffa* extracts exhibited dark-red and bright-red coloration, respectively, with a characteristic mild odor and an acidic taste. The purified *S. caudatum* extract was red, with a weakly characteristic odor and no distinctive taste.

Pharmaco-technical testing showed that all three dry extracts were hygroscopic. Flow behavior ranged from fairly good for lyophilized *H. sabdariffa* to extremely poor for purified *H. sabdariffa* and poor for purified *S. caudatum*; these deficiencies were improved by incorporating colloidal silica as a glidant.

The final formulation yielded capsules that were cylindrical, smooth, clean, and undeformed, with dark-red, red, or bright-red appearance depending on the extract. Extract fill masses were uniform, averaging 0.4344 g for lyophilized *H. sabdariffa*, 0.2259 g for purified *H. sabdariffa*, and 0.2755 g for purified *S. caudatum*, and each unit was designed to deliver 2.5 mg of active ingredient (anthocyanins). Capsules offer several well-recognized advantages—precise dosing and rapid gastric disintegration among them. The results presented here are experimental and provide a basis for further development.

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REFERENCES

1. BAKCHICHE B, GHERIB A. Activités antioxydantes des polyphénols extraits de plantes médicinales de la pharmacopée traditionnelle d'Algérie.
2. HMAMOUCHE M, LARROQUE M, MUNIER S, et al. Identification de polyphénols, évaluation de leur activité antioxydante et étude de leurs propriétés biologiques.
3. KABORE B. Anthocyanes, activités antioxydantes et traçabilité des extraits : cas des calices des fleurs de *Hibiscus sabdariffa* L. et des gaines des tiges de *Sorghum caudatum* H. Université Joseph KIZERBO ; 2019.
4. PALE E. Etude des anthocyanes des plantes du Burkina Faso : Structure et activités antioxydantes. Université de Ouagadougou ; 2002.
5. SEN S, CHAKRABORTY R, SRIDHAR C, et al. Free radicals, antioxidants, diseases and phytomedicines : current status and future prospect. 3.
6. SARDESAI VM. Role of Antioxidants in Health Maintenance. *Nutr Clin Pract* 1995; 10: 19-25. <https://doi.org/10.1177/011542659501000119> PMID:7898413
7. LATINO-MARTEL P, BACHMAN P. Nutrition chez le patient adulte atteint de cancer : compléments alimentaires antioxydants pendant et au décours du traitement des cancers. *Nutr Clin Métabolisme* 2012; 26: 238-246. <https://doi.org/10.1016/j.nupar.2012.10.008>
8. DEFRAIGNE JO, PINCEMAIL J. Stress oxydant et antioxydants : Rev Med Liège.
9. ATTA E, Mohamed N, ABDELGAWAD A. Antioxidants : An Overview on the Natural and Synthetic Types. *Eur Chem Bull* 2017; 6: 365-375. <https://doi.org/10.17628/ecb.2017.6.365-375>
10. KAROU D, DICKO MH, SIMPORE J, et al. Antioxidant and antibacterial activities of polyphenols from ethnomedicinal plants of Burkina Faso. *Afr J Biotechnol* 2005; 4: 823-828.
11. CISSE M, DORNIER M, SAKHO M, NDIAYE A, REYNES M, SOCK O. Le bissap (*Hibiscus sabdariffa* L.) : composition et principales utilisations. *Fruits*. mai 2009;64(3):179-93. <https://doi.org/10.1051/fruits/2009013>
12. BOUA R, Abdoul-latif F, O.H. K, et al. Phenolic compounds and antioxidant activity of 10 West African Sorghum Varieties.
13. RENAUD SC, GUEGUEN R, SCHENKER J, et al. Alcohol and mortality in middle-aged men from eastern France. *Epidemiol Camb Mass* 1998 ; 9: p 184-188. <https://doi.org/10.1097/00001648-199803000-00014>
14. HYARDIN A, CUNY A, MEJEAN L. La fonctionnalité alimentaire : illusion aujourd'hui, réalité demain. *Cahier Nutrition Diététique* 2007 ; 42: p 146-152. [https://doi.org/10.1016/S0007-9960\(07\)88757-8](https://doi.org/10.1016/S0007-9960(07)88757-8)
15. FAVIER A. Intérêt conceptuel et expérimental dans la compréhension des mécanismes des maladies et potentiel thérapeutique.
16. EVERSLEY TC. Mémoire présenté à la Faculté des études supérieures en vue de l'obtention du grade de M.Sc. en nutrition avec mémoire. 176 p.
17. Dr BEN MOUSSA. Département de pharmacie Batna. 6.

18. ADJEA F, LOZANO Y, ADIMA AA, MEUDEEC E, GAYDOU E, AGBO N'zi G. Structures et composition en anthocyanes d'extraits aqueux de plantes de Côte d'Ivoire *Delonix regia*, *Hibiscus sabdariffa* et *Carapa procera*. In Cirad; 2007
19. NGUYEN TT. Éco-extraction et encapsulation de pigments caroténoïdes et anthocyanes à partir de plantes tropicales. 159 p.
20. BEYE C, TOUNKARA LS, SECK MA, THONART P, FICKERS P. Opportunités pour la valorisation des végétaux riches en anthocyanes comme sources de colorants alimentaires (synthèse bibliographique). *Biotechnol Agron Soc Environ*. 2015;
21. GUILLOUTY Amandine. Plantes médicinales et antioxydants. Université Toulouse III Paul SABATIER, 2016.
22. GENEVEY L, SCHUTZ C. Législation du complément alimentaire et étude des compositions de deux types de compléments alimentaires. 184 p.
23. HEMA. A. Etude de molécules bioactives isolées de plantes du Burkina Faso. Thèse de doctorat unique Université de Ouagadougou. 170 p.
24. RIBEREAU-GAYON P. (168). Les composés phénoliques des végétaux. Ed. Dunod, Paris. 254 pages. Ed. Dunod, p. 168.
25. SAWADOGO O. Mise au point d'un complément alimentaire à base d'extraits secs des calices de fleurs de *Hibiscus sabdariffa* et de gaines des tiges de *Sorghum caudatum*. 2022 [cité 27 juin 2023].
26. E. PALE, M. Kouda-Bonafos, M. Nacro. Caractérisation et mesure des activités anti-radicalaires d'anthocyanes de plantes du Burkina Faso. *Comptes Rendus Chim*. oct 2004;7(10;11):973; 980. <https://doi.org/10.1016/j.crci.2003.12.019>
27. BARTHE J-M. Une réglementation pour les compléments alimentaires : quelles garanties pour le consommateur. 2009 ; 53.
28. Ratnam DV, Ankola DD, Bhardwaj V, Sahana DK, Kumar MNVR. Role of antioxidants in prophylaxis and therapy: A pharmaceutical perspective. *Journal of Controlled Release*. 20 juill 2006;113(3):189-207. <https://doi.org/10.1016/j.jconrel.2006.04.015> PMID:16790290
29. Codex A. Comité du codex sur les additifs alimentaires. 2011
30. Pharmacopée européenne. Assurance-qualité au préparatoire : maîtrise de la non-conformité. 5ème Edition. (5ème Edition).
31. Artemisia annua and Artemisia afra tea infusions vs. artesunate-amodiaquine (ASAQ) in treating *Plasmodium falciparum* malaria in a large scale, double blind, randomized clinical trial
32. OCDE. Pharmacopée européenne. Phytomedicine. 2008a; Tome1(6ème Edition):178-568.
33. DELEUIL M. Approche du comportement des poudres. Approche du comportement des poudres. 1987;(8):668-75.
34. Kabar K, Benlakehal I, Lefnaoui S. Développement galénique et contrôles physico-chimiques et biopharmaceutique d'une forme pharmaceutique sèche comprimée. Thèse 2020, consulté le 13 Aout 2023.
35. DJOKO E. International Journal of biological and Chemical Sciences. 2018 [cité 6 juin 2023]. Formulation d'un médicament traditionnel amélioré à visée antimicrobienne à base de *Euphorbia hirta* Linn. <https://doi.org/10.4314/ijbcs.v12i2.4>
36. Allo O, Blanc P, MA Dalmasso. Pharmacie galénique BP. Groupe Liaisons Santé. 2005;(2ème édition).
37. Rowe RC, Sheskey P, Quinn M. Handbook of Pharmaceutical excipients. Libros Digitales - Pharmaceutical Press ; 2009 [cité 13 août 2023].
38. Lieberman HA, Lachman L, Schwartz JB, éditeurs. Pharmaceutical dosage forms--tablets. 2nd ed., rev.expanded. New York: Dekker; 1989. 3 p.
39. Zhang Y, Law Y, Chakrabarti S. Physical properties and compact analysis of commonly used direct compression binders. *AAPS PharmSciTech*. 1 déc 2003;4(4):62. <https://doi.org/10.1208/pt040462> PMID:15198557 PMID:PMC2750655
40. BOUDENDOUNA AH. Méthodologie de la formulation d'une forme orale solide à libération prolongée