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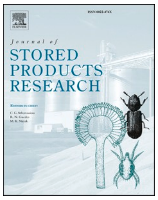

Physicochemical stability of aspalathin-rich rooibos iced tea powders under accelerated storage conditions as affected by formulation

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ABSTRACT

A stable, palatable, aspalathin-rich rooibos iced tea powder in a convenient single-serve format offers a viable functional beverage for reducing sugar intake. Our initial goal was to determine the optimal ratio of green rooibos extract (GRE) to fermented rooibos extract (FRE). This was essential to create a formulation that combined high aspalathin content with the sought-after sensory profile of traditional fermented rooibos. The second goal was to ascertain how the addition of common beverage ingredients (xylitol, citric, and ascorbic acid) and moisture (6 % and 53 % relative humidity, RH) affected the physicochemical stability of the mixtures during accelerated shelf-life storage (40 °C; 12 weeks). An FRE:GRE ratio of 1:0.5 was found to have a similar sensory profile as FRE, generally accepted by consumers, with a high aspalathin content (83.5 mg L^{-1}) compared to FRE alone (5.5 mg L^{-1}). During storage at 53 % RH, crystalline ingredients significantly decreased the color (lower L* and h°) and aspalathin (based on first-order reaction rate constants) stability. Changes in the crystal structure, affected by the interaction between ingredients, were observed using powder X-ray diffraction and Fourier-transform infrared spectroscopy. Minimal changes were observed for all parameters when storing the iced tea powders at 6 % RH, regardless of formulation. Ready-to-reconstitute aspalathin-rich rooibos iced tea powders should be stored in moisture-impermeable packaging to ensure a physically (including color) and chemically stable product.

1. Introduction

Aspalathin, a *C*-glucosyl dihydrochalcone unique to the *Aspalathus* genus, is the most important bioactive of rooibos (*Aspalathus linearis* (Burm.f.) R.Dahlgren) herbal tea (Joubert and De Beer, 2011). The “unfermented” green product contains particularly high levels of aspalathin, which has been extensively studied for its role in mediating the metabolic syndrome (Chaudhary et al., 2021; Muller et al., 2022).

A green rooibos ready-to-reconstitute (RTR) iced tea powder mixture in a single-serve format is a convenient dietary vehicle to supply aspalathin. An RTR product would be preferable to a ready-to-drink (RTD) product, as aspalathin oxidizes rapidly in solution. Depending on the formulation, aspalathin decreased by 22–33 % during ~3 months of storage at 25 °C in a green rooibos RTD beverage (De Beer et al., 2012), while 34–50 % degradation was observed for green rooibos RTR mixtures, protected from moisture uptake, over 12 months of storage at 25 °C (Human et al., 2021).

Production of a green rooibos convenience functional beverage powder presents the challenge of achieving high aspalathin content, while maintaining an acceptable sensory profile. South African consumers prefer traditional “fermented” rooibos (Van Zyl, 2021; Viljoen et al., 2017) as the fermentation process changes the color to red-brown and leads to increased fruity and sweet-associated aromas and flavors (Joubert et al., 2025). Therefore, a pure green rooibos iced tea (Human et al., 2021) would likely have low market acceptance. To create a palatable functional rooibos beverage without artificial flavors, we

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investigated formulations containing fermented rooibos extract (FRE) for its flavor and green rooibos extract (GRE) for its aspalathin content.

Formulating an RTR rooibos-based functional product intended to alleviate metabolic syndrome entails several key considerations. A pre-packed single-serve convenience product should be formulated so that the consumer only needs to add water. Such products require a carrier to aid in extract handling and bulking. Maltodextrin, the carrier commonly used by industry, is not suitable for diabetics as it is rapidly metabolized to glucose, resulting in a rapid increase in post-prandial blood glucose levels (Hofman et al., 2016). Inulin presents a better alternative: it is indigestible to humans, supports the growth of beneficial intestinal bacteria and the immune system, improves the blood lipid profile (Shoaib et al., 2016), and improves the wettability of spray-dried rooibos powder (Miller et al., 2018), important for reconstitution before consumption. This study will address knowledge gaps in developing a formulation with both an acceptable sensory profile and a high aspalathin content. It will also investigate how other food ingredients, such as xylitol, citric acid and ascorbic acid, affect the stability of aspalathin, other phenolic compounds and color.

The aims of the present study were to 1) determine the ratio of FRE to GRE for a beverage formulation with increased aspalathin content compared to FRE and improved sensory profile compared to GRE; and 2) assess the stability of the rooibos iced tea powder mixtures, in terms of physicochemical properties, as affected by formulation and relative humidity (RH) during accelerated storage.

2. Materials and methods

2.1. Materials and chemicals

Deionized water prepared using an Elix Advantage 5 UV system (Merck, Darmstadt, Germany) was further purified using a Milli-Q Reference A+ system (Merck) to obtain high-performance liquid chromatography (HPLC) grade water. HPLC gradient-grade acetonitrile and glacial acetic acid were from Merck. Food-grade ingredients were from Warren Chemicals (xylitol; Cape Town, South Africa), Brenntag (ascorbic acid; Johannesburg, South Africa), and Delite Dash Foods (citric acid hydrate and anhydrous citric acid; Cape Town, South Africa).

Spray-dried extracts of fermented (hot water extract; FRE) and green (aqueous ethanolic extract; GRE) rooibos were obtained from Afriplex (Paarl, South Africa). Inulin (Orafti high-performance inulin; 21–26 degree of polymerization, extracted from chicory root) was obtained from Savannah Fine Chemicals (Gardenview, South Africa). The two extracts were microencapsulated with inulin (1:1, m/m) by spray-drying using a Büchi B-290 mini spray-dryer (Büchi Labortechnik AG, Flawil, Switzerland), equipped with a dehumidifier and an oil-free air compressor (Haug Kompressoren AG, St. Gallen, Switzerland). The spray-drying parameters were as follows: spray nozzle tip diameter, 0.7 mm; atomizing air flow rate, 1744 L h^{-1}; atomizing air pressure, 6 bar; aspiration rate, 100 %; inlet temperature, 220 °C; outlet temperature, 95–105 °C; dehumidifier temperature, <5 °C; feed concentration 100 g L^{-1} soluble solids; feed flow rate, 0.63 L h^{-1}; nozzle cleaning frequency 4 min^{-1} (Miller et al., 2018). The feed solution was prepared using hot water (~90 °C) and stirred continuously using a magnetic stirrer to prevent solids from settling. The microencapsulated fermented and green rooibos extracts were coded FRE_IN and GRE_IN, respectively.

Authentic reference standards were sourced from Extrasynthese (Genay, France; isoorientin (99 %), orientin (99 %), isovitexin (99 %), vitexin (99 %), hyperoside (98 %), luteoloside (98 %)), Merck (isoquercitrin (>90 %)), Transmit (Gieβen, Germany; rutin (98 %)), and Phytolab (Vestenbergsgreuth, Germany; aspalathin (91 %)).

2.2. Descriptive sensory analysis (DSA) of iced tea formulations with varying amounts of green rooibos extract

Ethical approval was obtained for the involvement of human subjects (Stellenbosch University Research Ethics Committee for Social, Behavioral and Education Research; reference number 21996; approved on May 3, 2021), and informed consent was obtained from each subject prior to their participation in the study.

The iced tea formulations for DSA contained 1 g L^{-1} citric acid and 40 g L^{-1} sugar, similar to standard iced tea products on the market. Formulations A-E each contained FRE at 1.750 g L^{-1}. GRE was added at 0, 0.438, 0.876, 1.313, or 1.750 g L^{-1} to yield FRE:GRE ratios of 1:0, 1:0.25, 1:0.5, 1:0.75, and 1:1, respectively. Formulation F contained GRE only at 1.750 g L^{-1} (FRE:GRE 0:1).

A sensory panel ($n = 10$) experienced in the analysis of rooibos herbal teas was trained using a combination of the consensus and ballot methods (6 × 1 h sessions) (Lawless and Heymann, 2010). The panel generated sensory attributes for the iced teas using the protocol of Koch et al. (2012). Definitions for the selected attributes were compiled into a lexicon with references (Table S1; supporting information) to align assessors. Unsalted water biscuits and deionized water were used as palate cleansers.

Four independent batches of each formulation were prepared and assessed on different days. Blind-coded samples were served at room temperature (~21 °C) in black ISO wine-tasting glasses (~100 mL per glass) covered with plastic lids. Samples were served in a random order. Attribute intensities were rated on an unstructured line scale (0 = none; 100 = extremely high) using Compusense20®software (Compusense®, Guelph, Canada). Individual booths (light- and temperature-controlled; ~21 °C) were assigned to each assessor.

2.3. Storage stability testing of iced tea powder formulations

The composition of the iced tea powders for the storage stability experiment was varied to include the extracts spray-dried with inulin alone (FRE_IN (F1) and GRE_IN (F2)), the mixture of the two at a set ratio (FRE_IN:GRE_IN 1:0.5, m/m (F3)), F3 with xylitol (F4), F4 with citric acid hydrate (F5), F4 with anhydrous citric acid (F6) and F6 with ascorbic acid (F7). In this experiment, xylitol was used as a sugar substitute due to its non-caloric nature. The composition of the iced tea powders (g 100 g^{-1}) and the reconstituted iced teas (g L^{-1}) is shown in Table 1. Food-grade xylitol, citric acid hydrate, anhydrous citric acid, and ascorbic acid were finely milled using a ball mill. Three independent batches of each formulation were prepared by weighing the individual ingredients and thoroughly mixing them with a mortar and pestle.

Stability testing of the iced tea powders took place in a forced-air oven at 40 °C in sealed mini-hygrostats consisting of amber 24 mL vials containing the sample, a spring, and a glass insert with 200 μL of a saturated salt solution sealed with a Teflon-lined screw cap as described previously (Human et al., 2023). The temperature for accelerated storage was selected based on the ICH guidelines for pharmaceutical products (ICH Q1A(R2), 2003). Saturated solutions of KOH and NaBr were used to obtain 6 % and 53 % RH, respectively (Greenspan, 1977). For each replicate of each formulation, separate vials containing 80 mg of iced tea powder were prepared for each RH and time point. At each time point (0, 1, 2, 3, 4, 6, 8, 10, 12 weeks), the vials were removed from the oven, the glass inserts removed, and the powders reconstituted to the required concentration (Table 1) by sonication (5 min) using 10 % (v/v) aqueous DMSO.

2.4. Analysis of iced tea powder mixtures and ingredients

The water activity (a_w) of iced tea powders at $t = 0$ weeks was determined at 25 °C using a LabMaster-aw electric hygrometer (Novasina AG, Lachen, Switzerland). The moisture content of these samples was determined gravimetrically after drying at 100 °C for 1 h in a forced-air oven followed by 70 °C for 16 h in a vacuum oven. Values were expressed as g moisture per 100 g powder, dry basis (db).

A Q5000 Vapor Sorption Analyzer (TA Instruments, New Castle, DE, USA) was used for moisture sorption analysis of the iced tea powders

Table 1
Formulation of rooibos iced tea powder mixtures (g 100 g^{-1} powder mixture; reconstituted concentration (g L^{-1}) in brackets).

Formulation	FRE_IN	GRE_IN	Xylitol	Citric acid hydrate	Anhydrous citric acid	Ascorbic acid	Reconstituted concentration (g L^{-1})
F1	100 (3.50)	–	–	–	–	–	3.50
F2	–	100 (3.50)	–	–	–	–	1.750
F3	66.7 (3.50)	33.3 (1.75)	–	–	–	–	5.25
F4	7.7 (3.50)	3.9 (1.75)	88.4 (40.00)	–	–	–	45.25
F5	7.6 (3.50)	3.8 (1.75)	86.5 (40.00)	2.2 (1.00)	–	–	46.25
F6	7.6 (3.50)	3.8 (1.75)	86.5 (40.00)	–	2.2 (1.00)	–	46.25
F7	7.5 (3.50)	3.8 (1.75)	86.1 (40.00)	–	2.2 (1.00)	0.4 (0.20)	46.45

and their individual ingredients at 25 °C at $t = 0$ weeks. Before each analysis, the samples were dried at 60 °C for 1 h or until a constant weight was obtained (<0.0001 % fluctuation). The moisture sorption isotherm (MSI) data for the first adsorption run from 10 to 65 % RH were fitted to the Guggenheim–Anderson–de Boer (GAB) model as described by Peleg (2020).

Differential scanning calorimetry (DSC), X-ray powder diffraction (XRPD) and Fourier-transform infrared (FTIR) analyses were done on the iced tea powders at $t = 0$ weeks and $t = 14$ weeks at 40 °C/6 % RH and 40 °C/53 % RH, as well as their individual ingredients at $t = 0$ weeks. A Linseis Chip-DSC 1 calorimeter (Linseis, Selb, Germany) equipped with Linseis Smart software was used for DSC analysis. The samples (~2 mg) were placed in aluminum pans and sealed with an aluminum lid using a Schmidt® Technology crimping tool (St. Georgen, Germany). Dry nitrogen gas purge was performed at 20 mL min^{-1} and a heating rate of 10 °C min^{-1} was used. A Bruker D8 Advance powder X-ray diffractometer (Karlsruhe, Germany) set at 40 kV and 40 mA was used for XRPD analysis. A diffraction range of 4–40° 2θ and a scan rate of 0.1° min^{-1} were used. A PerkinElmer Spectrum 400 FTIR spectrometer equipped with a diamond attenuated total reflectance crystal and spectrum software (version 6.3.5.017) was used for the FTIR analysis. A scan resolution of 2 cm−1 over 650–4000 cm−1 was used.

2.5. *Analysis of reconstituted iced teas*

Color measurements (CIE L*, a*, and b* values) of reconstituted iced teas in 10 mm path-length polystyrene cuvettes were performed in triplicate using a CM-5 spectrophotometer in transmittance mode (Konica Minolta Sensing Inc., Tokyo, Japan). The 10° observer and D65 light source were used, and the hue angle and chroma values were calculated by the instrument software. ΔE values were calculated as follows: $\Delta E = [(L^*_2 - L^*_1)^2 + (a^*_2 - a^*_1)^2 + b^*_2 - b^*_1)^2]^{1/2}$

Aliquots of the reconstituted iced teas were frozen (−20 °C) until HPLC-diode array detection (DAD) analysis. After defrosting, ascorbic acid was added (~9 mg mL^{-1} final concentration) and the mixtures filtered (0.22 μm hydrophilic PVDF, Merck) before HPLC analysis as previously described by Walters et al. (2017). The Agilent 1200 HPLC comprised a 1290 quaternary pump with an in-line degasser, a 1200 series autosampler, column thermostat and diode array detector, all controlled by OpenLab software (Agilent, Santa Clara, CA, USA). The separation details are as follows: column, Agilent Poroshell 120 C18 2.7 μm, 150 × 4.6 mm; column temperature, 44 °C; mobile phase A, 2 % acetic acid (v/v); mobile phase B, acetonitrile; flow rate, 1 mL min^{-1}; detection, 288 (PPAG, aspalathin and nothofagin) and 350 nm (all other compounds). PPAG and nothofagin were quantified using their response factors against aspalathin (Walters et al., 2017). Stock solutions of the calibration standards (~1 mg mL^{-1} in DMSO; stored at −20 °C) were defrosted and diluted with water before analysis. The diluted mixtures, containing ~9 mg mL^{-1} ascorbic acid, were filtered through 0.22 μm hydrophilic PVDF filters (Merck).

2.6. *Statistical analysis*

DSA data were subjected to statistical analyses to confirm panel reliability and normality using a model including assessor and sample effects (Næs et al., 2010). The Shapiro-Wilk test indicated outliers (standardized residuals >3) that were removed. One-way univariate analysis of variance (ANOVA) was conducted on sample means over assessors using the GLM procedure of SAS software. Fisher's LSD was calculated for $p = 0.05$ to compare treatment means, with p values < 0.05 considered to be significant. Principal component analysis (PCA), using the correlation matrix, was performed using XLSTAT (2024.2.2) (Lumivero, 2024).

A completely random ANOVA with formulation as the main factor and sampling time as a repeated measures factor was performed on all variables for each RH level separately to compare formulations over time. Normality was assessed using the Shapiro-Wilk test, and Fisher's least significant difference ($p = 0.05$) was used to compare means for significant effects.

Kinetic modeling was performed for selected compounds in iced tea powders stored at 40 °C/53 % RH. Kinetic models (zero-, first-, and fractional order) were applied to each replicate set of each formulation, using the NLIN procedure in SAS. Model suitability was assessed using adjusted coefficients of determination (R^2_{adj}) and parity plots comparing estimated and measured concentrations. A completely random ANOVA was performed on the regression parameters to compare formulations, using the GLM procedure of SAS, following normality assessment (Shapiro-Wilk). Fisher's least significant difference ($p = 0.05$) was used to compare means for significant effects.

3. Results and discussion

3.1. *Sensory profiles for rooibos iced tea formulations*

DSA was conducted on rooibos iced tea formulations with varying fermented and green rooibos extract content to identify one with a sensory profile similar to fermented rooibos, but with a higher aspalathin content. The aroma lexicon was based on previous studies on rooibos iced tea (Human et al., 2021) and infusions (De Beer et al., 2024; Du Preez et al., 2020) (Table S1). The PCA bi-plot (Fig. 1) had a total explained variance of 86.5 %. Formulation A-E is associated with typical fermented rooibos attributes ('rooibos-woody' and 'honey' aroma and flavor; 'apricot' and 'citrus' aroma) (Jolley et al., 2017), as well as 'marmalade' aroma and flavor, on the left side of the plot. This association was strongest for A (FRE only). In contrast, formulation F (GRE only) strongly associated with typical green rooibos attributes ('tobacco', 'cooked oats', 'cardboard', and 'hay/dried grass' aroma and flavor) (De Beer et al., 2024) along with 'putty/rubber' aroma and flavor, 'citrus' flavor, and astringent mouthfeel on the right side of the plot. The ANOVA results confirmed that formulations A, B, and C had higher ($p < 0.05$) 'rooibos-woody' aroma and flavor intensities than the other formulations, with F having lower ($p < 0.05$) intensities than the other formulations (Table S2). The intensities of the primary fermented rooibos sensory attributes, i.e., 'rooibos-woody' and 'honey', and secondary attributes 'apricot', 'citrus' and 'marmalade' aroma and flavor notes (Jolley et al., 2017), were highest for A and progressively decreased with increasing green rooibos extract content, with F scoring lower ($p < 0.05$) than the others. 'Tobacco', 'hay/dried grass', and 'putty/rubber' aroma and flavor, and astringent mouthfeel intensities showed the opposite trend, while 'cooked oats' and 'cardboard' aromas were only perceived

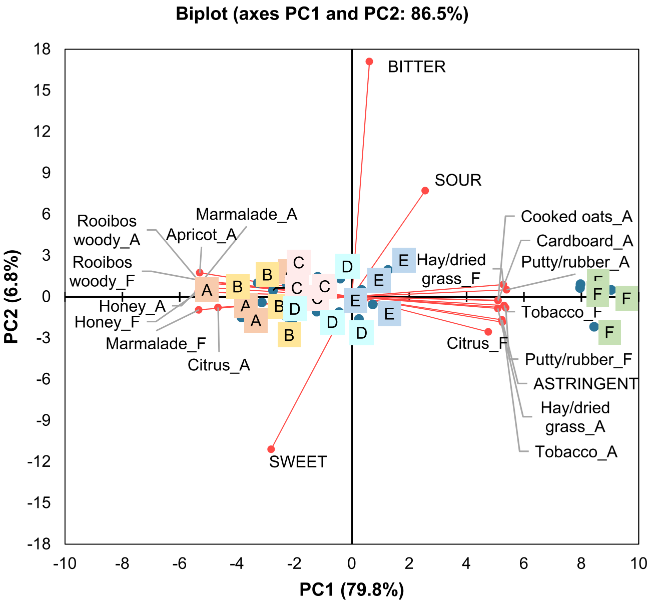

Fig. 1. Principal component analysis bi-plot illustrating the association between sensory attributes and rooibos iced tea samples containing varying concentrations of fermented and green rooibos extract, 1 g L^{-1} citric acid and 40 g L^{-1} sugar. Formulations A-E contained 1.750 g L^{-1} fermented rooibos extract. Formulation B-F contained 0.438, 0.876, 1.313, 1.750, and 1.750 g L^{-1} green rooibos extract. A and F at the end of each descriptor refer to aroma and flavor, respectively. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

in F. Sweetness and sourness remained comparable across formulations, although A was perceived as sweeter and less sour than F ($p < 0.05$).

Formulation C (FRE:GRE ratio 1:0.5) was selected for further investigation since its sensory profile was similar to that of formulation A, but with a much higher aspalathin concentration (83.5 vs 5.5 mg L^{-1}). Previously, commercial iced teas from South Africa were found to contain much lower aspalathin concentrations, ranging from not detected to 0.7 mg L^{-1}, than formulation A, potentially indicating poor product stability (Joubert et al., 2009).

3.2. *Storage stability of rooibos iced tea powder formulations*

RTR rooibos iced teas for the accelerated stability study were formulated to determine the effects of extract type (fermented only (F1), green only (F2) and a blend of fermented and green extracts (F3)) and commonly used iced tea ingredients, namely xylitol (low-kilojoule sweetener; F4-7), citric acid (acidity regulator; F5-7) and ascorbic acid (antioxidant; F7). Both citric acid hydrate (F5) and anhydrous citric acid (F6) were included, as a previous study (Human et al., 2021) showed that citric acid hydrate may release water and compromise aspalathin stability in green rooibos iced tea powders during storage.

3.2.1. *Moisture content, water activity (a_w) and moisture sorption isotherms (MSI)*

F1-3, containing only rooibos extract, had moisture contents between 1.8 and 2.1 g 100 g^{-1} db, while those containing crystalline ingredients (F4-7) had very low moisture content (<1 g 100 g^{-1} db) (Table 2). The water activity (a_w) values ranged from 0.082 to 0.125 for F1-3, while a_w increased to 0.224–0.239 for F4-7. Generally, an a_w of below 0.61 is considered sufficient to inhibit microbial, yeast, or mold growth (Fontana, 2000); however, its effect on physical stability is product-specific. All formulations had a_w values well below 0.61, indicating their microbial stability.

MSIs describe the process wherein water molecules are sorbed in a food system as a function of environmental RH, which can be used to predict product behavior during shelf life (Caballero-Cerón et al., 2015). The MSIs for F1-3 can be classified as Type II, typical of soluble materials, while those of F4-7 resemble Type III, typical of high-sugar foods (Fig. 2) (Caballero-Cerón et al., 2015). This was expected as F4-7 contains 86.1–88.4 g 100 g^{-1} xylitol, a sugar alcohol. All formulations exhibited marked hysteresis after the first adsorption cycle, indicated by different desorption and second adsorption patterns. This indicates that exposure of the formulations to high environmental RH would cause irreversible phase and structural changes, detrimental to their physical stability (Labuza and Altunakar, 2020). Therefore, low RH storage is recommended for all formulations.

Table 2

Moisture content (mean ± SD), water activity (a_w; mean ± SD), and monolayer moisture content (M_0) for the Guggenheim–Anderson–de Boer (GAB) model fitted to moisture adsorption data obtained at 25 °C for rooibos iced tea powder formulations[a].

Formulation	Moisture content (g 100 g^{-1} dry basis)	Water activity (a_w)	Monolayer moisture content (M_0) (g 100 g^{-1} dry basis)
F1	1.77 ± 0.64	0.082 ± 0.015	1.25
F2	2.06 ± 0.38	0.123 ± 0.006	0.85
F3	2.12 ± 0.55	0.125 ± 0.008	1.24
F4	0.36 ± 0.14	0.239 ± 0.045	0.14
F5	0.61 ± 0.09	0.233 ± 0.052	0.27
F6	0.44 ± 0.11	0.224 ± 0.048	1.81
F7	0.43 ± 0.11	0.233 ± 0.048	0.56

[a] Composition of formulations is given in Table 1.

Moisture adsorption was significantly lower for F4-7 than for F1-3 at the same RH. The bulk of F4-7 is xylitol, which exhibited a moisture content of <0.2 g 100 g^{-1} at 90 % RH (data not shown). This indicates that the addition of food-grade crystalline ingredients could potentially increase the stability of rooibos iced tea powders by decreasing moisture adsorption under high humidity. Ascorbic and citric acid addition (F5-7) slightly increased the moisture uptake compared to F4 (Fig. 2). This is due to the slightly higher RH_0 values (environmental RH where deliquescence occurs) of the acids (except the anhydrous citric acid) compared to xylitol.

The GAB models showed good coefficients of determination (R^2, Table S3) for the experimental and predicted MSI data. Visual inspection (Fig. S1) and the mean absolute errors between the experimental and predicted values (Table S3) indicated an excellent fit for the GAB model. $M_{0,}$ defined as a single layer of moisture adsorbed in the pores and capillaries of a solid, is thus unavailable for chemical reactions. When the moisture content of a formulation exceeds its M_0, the excess moisture is considered reactive and may compromise product stability (Caballero-Cerón et al., 2015; Mauer, 2020). Based on this explanation, both F6 and F7 with moisture contents below their respective M_0 values can be considered the formulations with the highest potential stability during storage in terms of moisture-driven reactions (Table 2).

3.2.2. *Crystal structure and phase transformation during storage (DSC and XRPD)*

Fig. 3 and S3 show the XRP diffractograms and DSC thermograms, respectively, for the iced tea powders at t = 0 weeks and t = 14 weeks. The DSC thermogram of citric acid hydrate displayed a clear endothermic peak at 50–70 °C, indicating the presence of bound water (Fig. S2), which was absent in the thermogram for anhydrous citric acid. F1-3, containing only rooibos extract and inulin, were amorphous as indicated by the broad halo and the absence of sharp peaks in their XRP diffractograms. These powders showed evidence of crystallization after storage at 40 °C/53 % RH, evidenced by the formation of sharp crystalline peaks protruding from the amorphous halo structure in the XRP diffractograms. The XRP diffractograms and DSC thermograms for F4-7 resemble those of their crystalline ingredients, with xylitol-associated peaks the most prominent. After storage, the XRP diffractogram

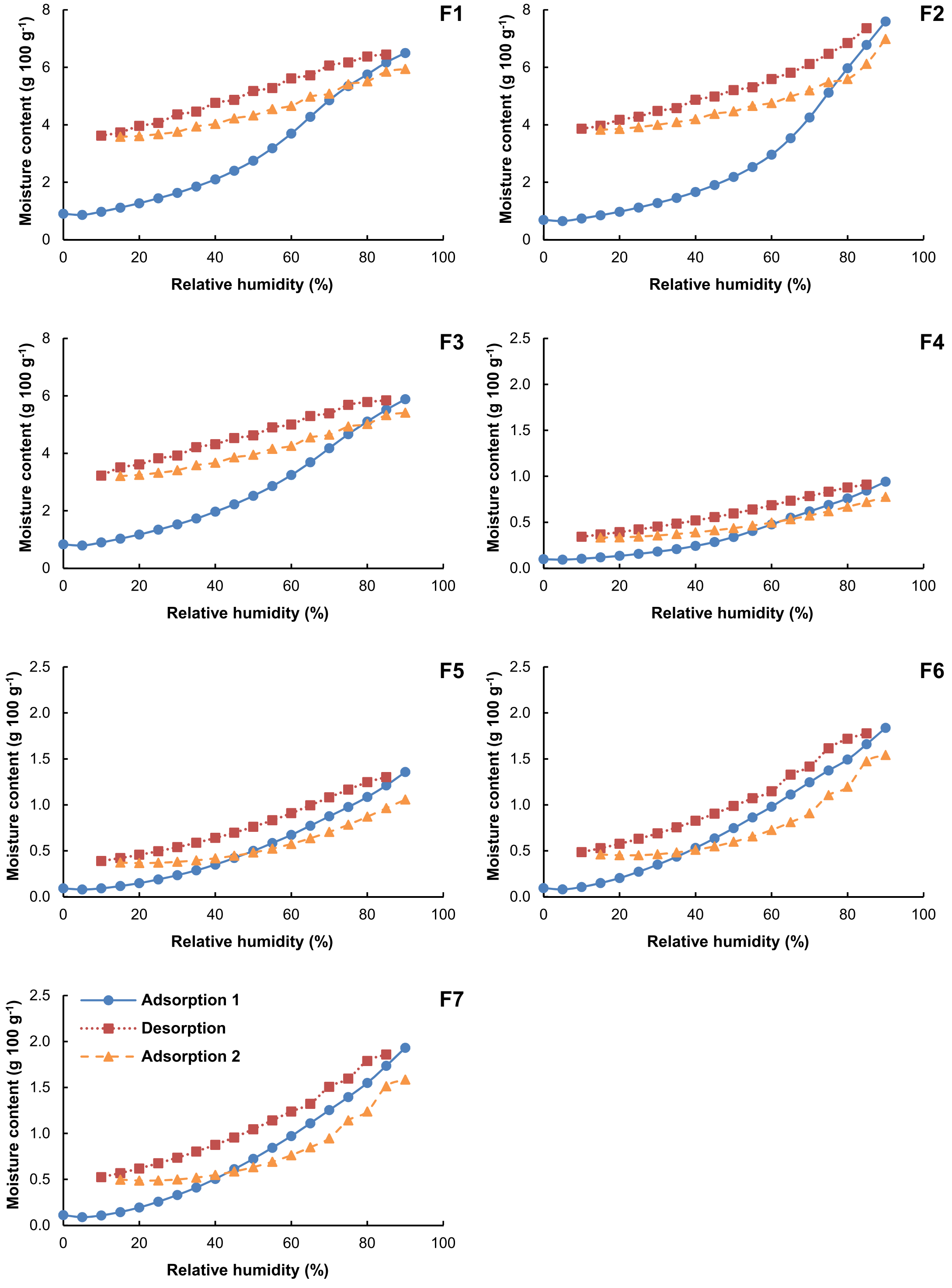

Fig. 2. Moisture sorption isotherms for rooibos iced tea powder formulations. The composition of the formulations is given in Table 1. Moisture content is on a dry basis.

showed broadening, intensity changes, and peak merging, reflecting physical transformations. These changes aligned with the pronounced hysteresis observed in the MSIs, indicating permanent physical change upon moisture exposure (Fig. 2). In the DSC thermograms, changes in peak intensity and a shift in the peak associated with the melting of xylitol were also observed.

Amorphous and crystalline solids respond differently to moisture. Amorphous materials can crystallize, as shown by the XRP diffractograms for rooibos extract-inulin mixtures. At high RH values, water absorption lowers the glass transition temperature (T_g), due to increasing molecular spacing and mobility (Mauer, 2020). This results in physical collapse and water desorption, followed by crystallization. Crystalline solids, such as xylitol, citric and ascorbic acid, undergo deliquescence when the environmental RH exceeds their RH_0. This partial solvation, occurring in equilibrium with desolvation, can promote the formation of liquid bridges, recrystallization, and co-crystallization with entrapped water and other crystalline solids. In mixtures containing multiple crystalline and amorphous ingredients,

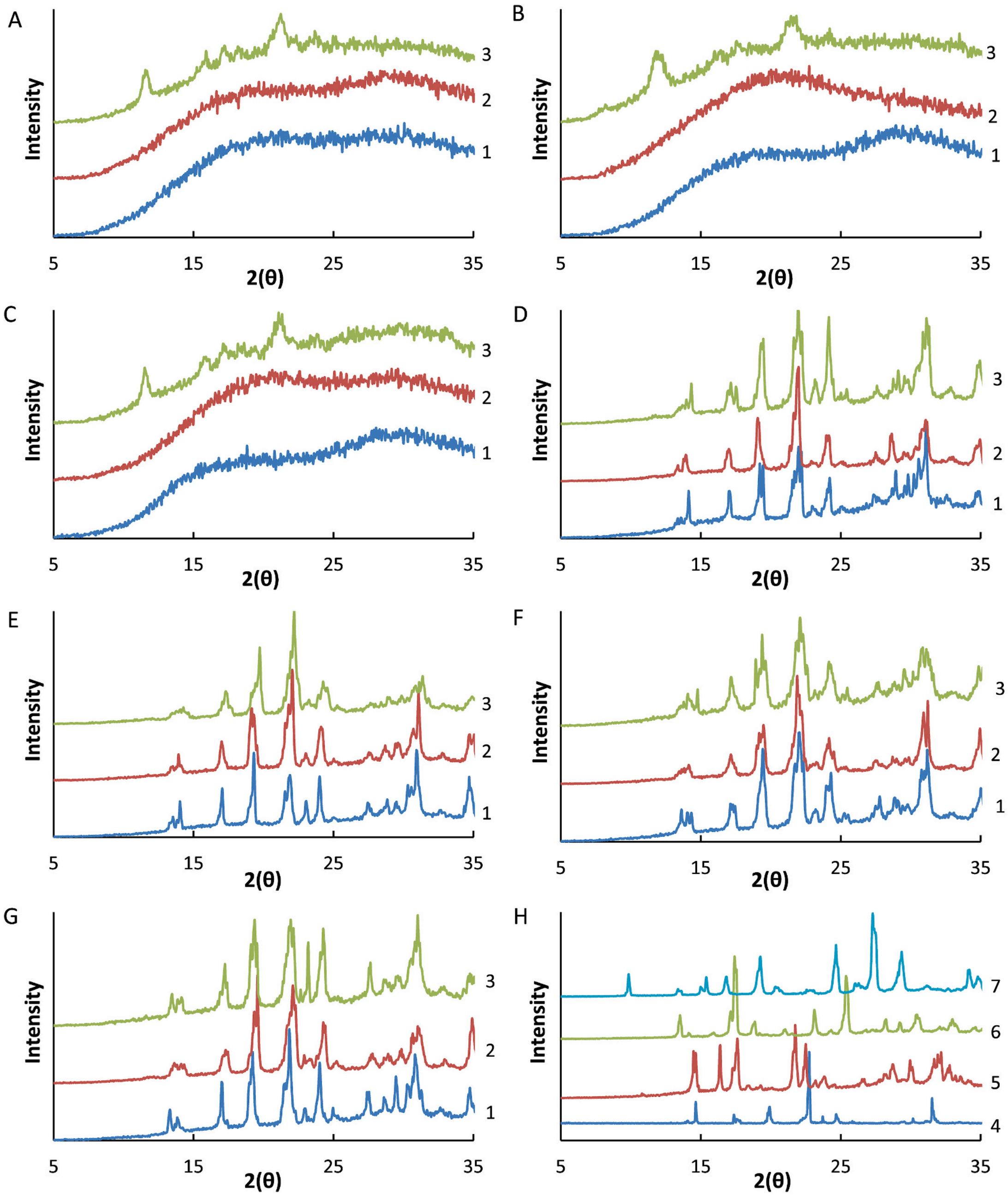

Fig. 3. X-ray powder diffractograms for powder mixtures (A) F1, (B) F2, (C) F3, (D) F4, (E) F5, (F) F6 and (G) F7 (1) before storage and stored at (2) 40 °C/8 % RH and (3) 40 °C/75 % RH. (H) Shows the individual ingredients used to prepare the mixtures: (4) xylitol; (5) citric acid hydrate, (6) citric acid anhydrous, and (7) ascorbic acid. The composition of the formulations is given in Table 1.

mutual deliquescence and water absorption can further increase the sensitivity of a powder formulation to moisture (Voelker et al., 2020). These changes in the physical structure of both crystalline and amorphous solids can result in unwanted stickiness and caking of powder formulations (Stoklosa et al., 2012).

3.2.3. Chemical interactions and changes during storage (FTIR)

The FTIR spectra (Fig. S3) for the powder mixtures before and after storage, as well as their individual ingredients, indicate changes in the chemical interactions between components. After storage, the rooibos-inulin mixtures (F1-3) showed reduced peak intensities at ~1600 cm^{-1} and ~1025 cm^{-1}, representing carboxyl and aromatic moieties (Pavia et al., 2015) abundant in the phenolic compounds of the rooibos extracts. These changes, most pronounced in samples stored at 40 °C/53 % RH, indicate either chemical degradation or water uptake accompanied by hydrogen bonding between the absorbed water molecules and carboxyl moieties. The spectra of the mixtures containing crystalline ingredients (F4-7) closely resembled that of xylitol, their main component. After storage, minimal spectral changes were observed, except for F4, which showed a larger and deformed peak at 3500-3000 cm^{-1}. This peak is linked to the hydroxyl moiety of xylitol, and its deformation also indicates water absorption and subsequent hydrogen bonding. A change in hydroxyl and carboxyl moieties has been linked to the amount of sorbed water and hydrogen bonding, as shown for natural fiber composites (Célino et al., 2014).

3.2.4. Changes in color during storage

The CIELab color parameters of the reconstituted iced teas were measured to assess changes in the powder color, as direct measurement of the powder samples (only 80 mg each) was not possible. Formulations containing citric acid hydrate and anhydrous citric acid (F5 and F6) had very similar color changes (Table S4); therefore, only F6 is discussed (Fig. 4). The CIELab color parameters of reconstituted iced teas changed substantially during storage at 53 % RH compared to 6 % RH (Fig. 4; Table S4). At the low RH value, L* and h° values remained stable ($p \geq 0.05$) over 14 weeks, except for h° of F2 (GRE_IN only), and most ΔE values after storage were <3. At 53 % RH, L* decreased significantly ($p < 0.05$) only for F6 and F7, while h° decreased significantly ($p < 0.05$) for F4-6, indicating a shift from very light yellow-orange to a darker, more orange hue. While the addition of crystalline ingredients (xylitol, citric, and ascorbic acid) had a minimal effect on the initial color of the reconstituted iced teas, they reduced the color stability during storage at 53 % RH, shown by higher ΔE values after storage for F6 (15.50) and F7 (12.98) compared to F4 (5.14). A darkening and change to orange/brown were also previously shown for green rooibos powder mixtures, particularly for mixtures with crystalline ingredients at higher temperatures and 65 % RH (Human et al., 2021). The more drastic visual changes observed in the latter study were due to longer storage and the exclusive use of green rooibos extract, which has a lighter initial color.

3.2.5. *Changes in phenolic composition during storage*

The phenolic composition of the iced tea powders showed relatively small changes (<20 %) during storage at 40 °C/6 % RH (Table S4). At 53 % RH, substantial losses in aspalathin and nothofagin (up to 80 % decrease), followed by PPAG (up to 58 % decrease), occurred (Fig. 5; Table S4). Minimal changes were observed for orientin, isoorientin, vitexin, and luteoloside (<20 %; Fig. S4), irrespective of formulation, and their data were, therefore, not modeled. The remaining phenolic compounds, showing notable changes during storage at 40 °C/53 % RH, were modeled (Fig. 5; Table 3). In most cases, the first-order or fractional conversion (based on the first-order model, but with non-zero equilibrium concentrations) reaction kinetic models gave the highest R^2_{adj} values (Table S5). Mostly, the differences in R^2_{adj} values for the two models were small, except for formulations where very little degradation was noted. In order to compare k-values between formulations and compounds, the first-order model was selected for all compounds.

The reaction rate constants (k) for the degradation of all the modeled compounds, except PPAG (Table 3), were significantly ($p < 0.05$) lower in powders containing only rooibos extracts spray-dried with inulin (F1-3). This indicates that these compounds were relatively stable in inulin-encapsulated extracts, irrespective of the extract or mixture. Similar results were found for aspalathin and nothofagin degradation in green rooibos extract spray-dried with inulin and stored in sealed vials (no moisture uptake, similar to low RH) or moisture-permeable packaging (moisture uptake similar to high RH) at 30 °C/65 % RH and 40 °C/65 % RH (Human et al., 2021). Similarly, phenolic compounds in a spray-dried green honeybush extract-inulin mixture (*C. subternata*) also demonstrated good stability (at 25 °C/60 % RH and 40 °C/75 % RH) in the absence of crystalline ingredients, in a similar packaging material (De Beer et al., 2018). Even though some phenolic degradation occurred in F1-3, losses were minor (Fig. 5). However, the addition of crystalline ingredients, i.e., xylitol, citric, and ascorbic acid, significantly ($p < 0.05$) accelerated the degradation of aspalathin and nothofagin. The k-values for PPAG degradation in F1-4 were significantly ($p < 0.05$) lower than those of the other formulations, with no significant differences ($p \geq 0.05$) between F5-7. This indicates that the PPAG degradation rate was unaffected by xylitol but was accelerated by the addition of citric acid (hydrate or anhydrous) and ascorbic acid.

No significant differences ($p \geq 0.05$) in the k-values were observed between formulations containing citric acid hydrate (F5) and anhydrous citric acid (F6) for any of the compounds, indicating that bound water in citric acid hydrate did not significantly impact compound stability (Table 3). The effect of citric acid on the degradation rate constants depended on the compound. Citric acid had a protective effect on aspalathin, as also observed by Human et al. (2021), and (*S*)-6-β-D-glucopyranosyleriodictyol (SE6G), as reflected by their

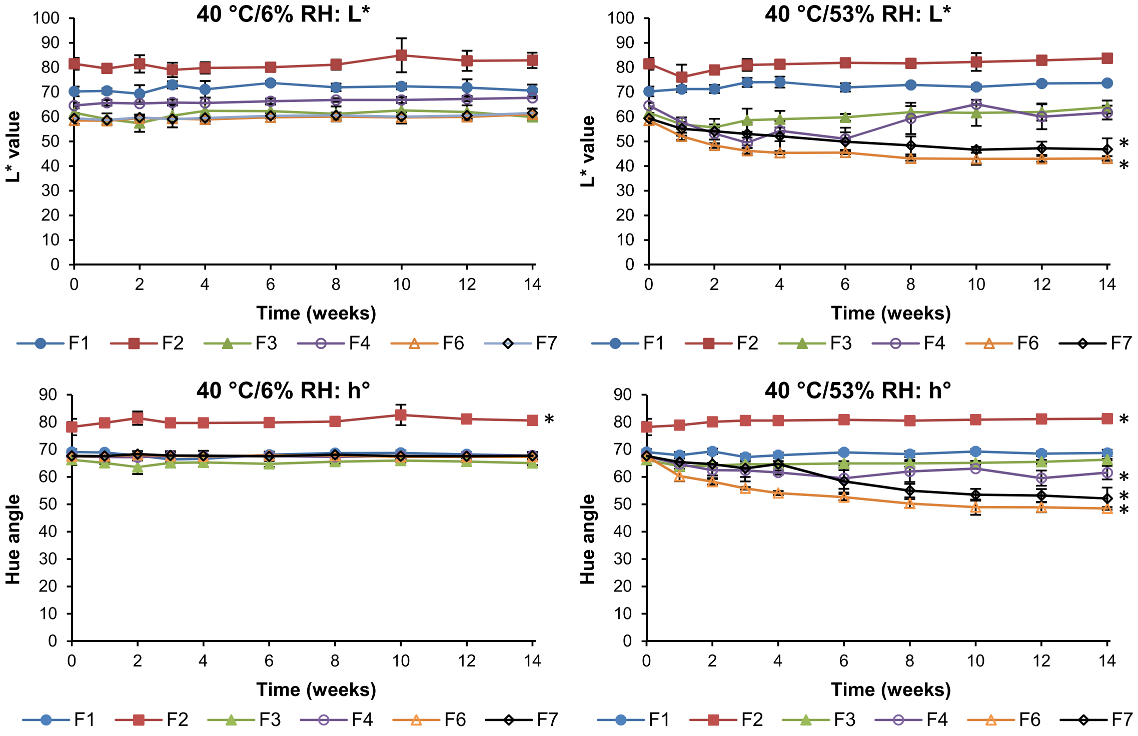

Fig. 4. Changes in CIELab color parameters (mean ± standard deviation) in reconstituted iced tea powder formulations stored at 40 °C/6 % relative humidity (RH) and 40 °C/53 % RH. Composition of formulations is given in Table 1. * indicates a significant difference ($p < 0.05$) between means for 0 and 14 weeks. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

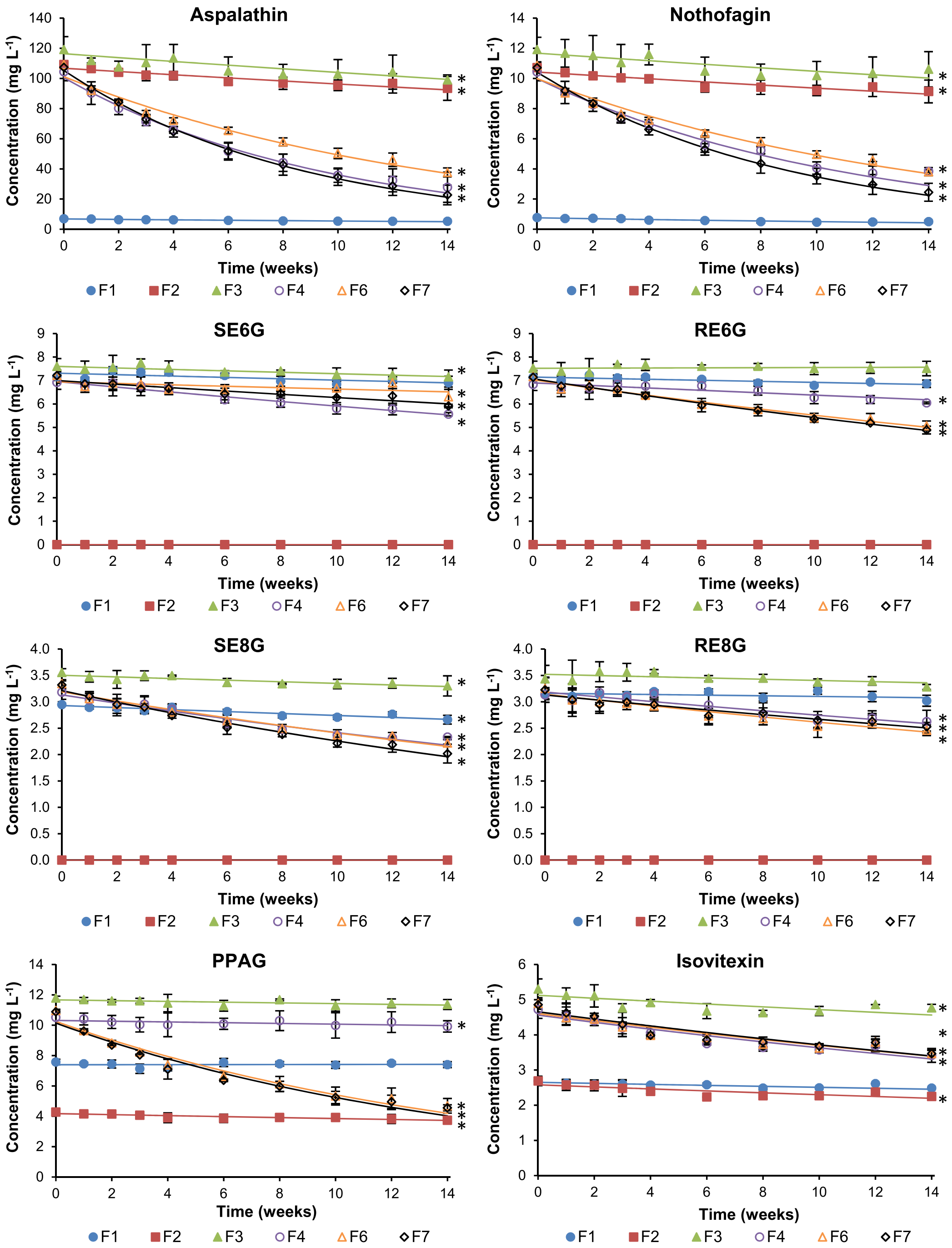

Fig. 5. Phenolic composition changes in reconstituted iced tea powder formulations stored at 40 °C/53 % relative humidity (RH). Markers indicate means ± standard deviation for measured data and lines indicate predicted compound concentration using first-order kinetic modelling. Composition of formulations is given in Table 1. * indicates a significant difference ($p < 0.05$) between means for 0 and 14 weeks.

Abbreviations: PPAG, *Z*-2-(β-D-glucopyranosyloxy)-3-phenylpropenoic acid; RE6G, (*R*)-6-β-D-glucopyranosyleriodictyol; RE8G, (*R*)-8-β-D-glucopyranosyleriodictyol; SE6G, (*S*)-6-β-D-glucopyranosyleriodictyol; SE8G, (*S*)-8-β-D-glucopyranosyleriodictyol.

significantly lower k-values for F5 and F6 ($p < 0.05$) compared to F4. The opposite effect was observed for PPAG, bioquercetin, hyperoside, and (*R*)-6-β-D-glucopyranosyleriodictyol (RE6G), while the k-values for other compounds did not differ significantly ($p \geq 0.05$) between F4, F5, and F6. Addition of ascorbic acid (F7) either significantly ($p < 0.05$) increased the k-values compared to F6 (aspalathin, nothofagin, bioquercetin, rutin, SE6G, and (*S*)-8-β-D-glucopyranosyleriodictyol (SE8G)) or had no significant ($p \geq 0.05$) effect. Comparable effects of ascorbic acid addition on phenolic degradation were also previously reported (De Beer et al., 2018; Human et al., 2021).

Previous research attributed accelerated degradation of phenolic compounds in iced tea powders containing green rooibos extract and citric acid hydrate stored in sealed vials to the release of bound water (Human et al., 2021). In the present study, most compounds showed higher % change values after storage at 40 °C/6 % RH in the presence of citric acid hydrate (F5) than anhydrous citric acid (F6) (Table S4).

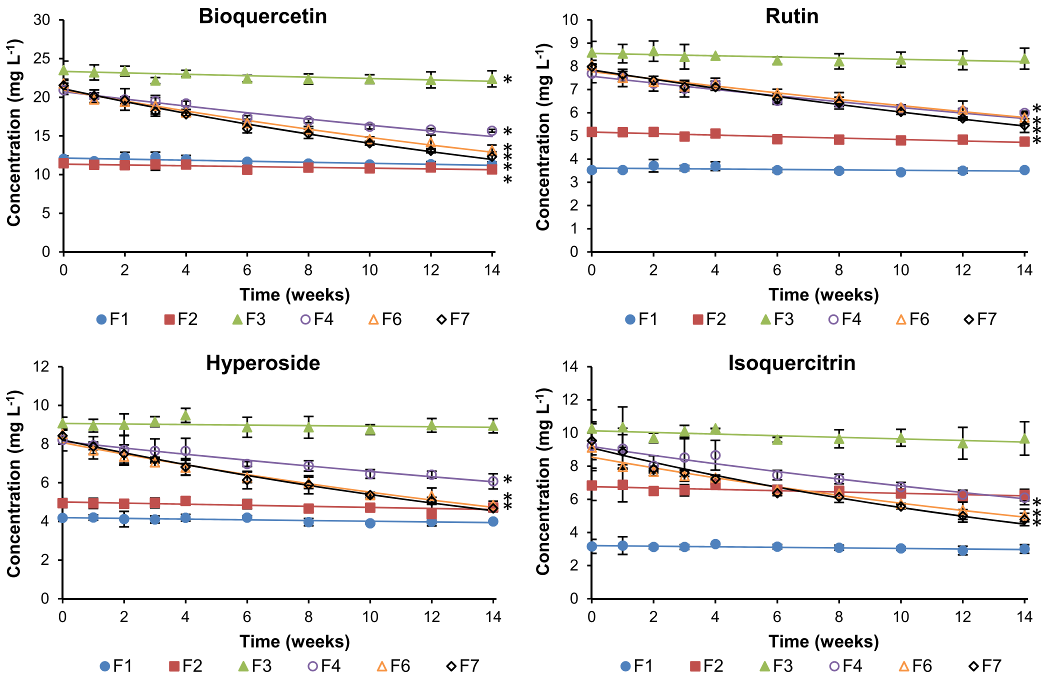

Fig. 5. (*continued*).

Table 3
Reaction rate constants (k; week^{-1}; mean ± SD, n = 3)[a] for degradation of selected phenolic compounds in rooibos iced tea powder formulations[b] during 14 weeks of storage at 40 °C and 53 % relative humidity.

Compounds	F1	F2	F3	F4	F5	F6	F7
Aspalathin	0.0224 c ± 0.0067	0.0103 c ± 0.0056	0.0113 c ± 0.0024	0.1024 a ±0.0269	0.0725 b ± 0.0057	0.0719 b ± 0.0067	0.1146 a ±0.0204
Nothofagin	0.0410 c ± 0.0259	0.0108 d ± 0.0055	0.0109 d ± 0.0022	0.0885 ab ± 0.0226	0.0721 b ± 0.0053	0.0713 b ± 0.0060	0.1098 a ±0.0176
PPAG	−0.0002 b ± 0.0007	0.0081 b ± 0.0038	0.0021 b ± 0.0012	0.0024 b ± 0.0024	0.0645 a ±0.0095	0.0638 a ±0.0096	0.0660 a ±0.0046
Isovitexin	0.0055 b ± 0.0009	0.0115 b ± 0.0040	0.0083 b ± 0.0026	0.0228 a ±0.0058	0.0232 a ±0.0023	0.0218 a ±0.0029	0.0225 a ±0.0036
Bioquercetin	0.0057 d ± 0.0014	0.0047 d ± 0.0002	0.0040 d ± 0.0005	0.0234 c ± 0.0050	0.0344 b ± 0.0021	0.0345 b ± 0.0019	0.0405 a ±0.0031
Hyperoside	0.0045 c ± 0.0031	0.0058 c ± 0.0080	0.0016 c ± 0.0056	0.0211 b ± 0.0061	0.0373 a ±0.0080	0.0382 a ±0.0079	0.0421 a ±0.0065
Rutin	0.0026 c ± 0.0017	0.0064 c ± 0.0008	0.0030 c ± 0.0005	0.0196 b ± 0.0055	0.0219 ab ± 0.0033	0.0210 b ± 0.0023	0.0263 a ±0.0025
Isoquercitrin	0.0055 b ± 0.0145	0.0061 b ± 0.0124	0.0050 b ± 0.0127	0.0300 a ±0.0122	0.0447 a ±0.0153	0.0394 a ±0.0055	0.0503 a ±0.0148
SE6G	0.0041 cd ± 0.0009	0 d[c]	0.0042 cd ± 0.0020	0.0164 a ±0.0035	0.0075 bc ± 0.0052	0.0045 cd ± 0.0044	0.0110 b ± 0.0015
RE6G	0.0032 c ± 0.0011	0 c[c]	−0.0004 c ± 0.0014	0.0079 b ± 0.0025	0.0247 a ±0.0035	0.0242 a ±0.0046	0.0266 a ±0.0022
SE8G	0.0067 c ± 0.0017	0 d[c]	0.0045 cd ± 0.0028	0.0260 b ± 0.0054	0.0284 b ± 0.0009	0.0280 b ± 0.0021	0.0353 a ±0.0032
RE8G	0.0019 b ± 0.0025	0 b[c]	0.0033 b ± 0.0033	0.0148 a ±0.0038	0.0193 a ±0.0060	0.0185 a ±0.0068	0.0159 a ±0.0068

Means with different alphabet letters are significantly different ($p < 0.05$).
Abbreviations: PPAG, *Z*-2-(β-D-glucopyranosyloxy)-3-phenylpropenoic acid; RE6G, (*R*)-6-β-D-glucopyranosyleriodictyol; RE8G, (*R*)-8-β-D-glucopyranosyleriodictyol; SE6G, (*S*)-6-β-D-glucopyranosyleriodictyol; SE8G, (*S*)-8-β-D-glucopyranosyleriodictyol.
[a] Fitted to the first-order reaction kinetics model.
[b] Composition of formulations is given in Table 1.
[c] Not detected.

However, the k-values for degradation were not significantly different ($p \geq 0.05$) (Table 3). At the higher RH level in this study, as well as in the previous study involving moisture-permeable sachets, this effect of citric acid hydrate was not observed, likely due to the already high humidity level. This explanation is also supported by studies on green tea extract powder formulations (Ortiz et al., 2008, 2009), which showed that citric acid accelerated catechin degradation at RH $\geq$ 58 %, but had no effect below this threshold. At RH $\geq$ 58 %, the T_g of the mixture was lower than the storage temperature. Without the addition of citric acid, the T_g of the green tea powder at 58 % RH was higher than the storage temperature, contributing to greater stability.

4. Conclusions

Blending fermented and green rooibos extracts yielded iced tea powder formulations that retained the desirable sensory properties of fermented rooibos while providing an elevated aspalathin content. Crystalline ingredients, specifically the low-kilojoule sweetener xylitol, acidity regulator citric acid, and antioxidant ascorbic acid, did not compromise the phenolic stability of the powder mixtures, highlighting their potential compatibility when moisture uptake is effectively controlled. However, exposure to moisture substantially reduced overall powder stability, with the most pronounced changes observed for formulations containing xylitol, citric acid, and ascorbic acid. The dihydrochalcones, aspalathin and nothofagin, were the least stable of the quantified compounds. Greater phenolic degradation was consistently associated with more pronounced physical and color changes in the reconstituted iced teas. Therefore, to retain both phenolic integrity and overall physicochemical stability, ready-to-reconstitute functional rooibos iced tea formulations should be protected from moisture uptake by storage in moisture-impermeable packaging.

CRediT authorship contribution statement

Dalene de Beer: Writing – original draft, Visualization, Resources, Investigation, Funding acquisition, Conceptualization. **Chantelle Human:** Writing – review & editing, Investigation. **Brigitte V.P. du Preez:** Writing – review & editing, Investigation. **Erika I. Moelich:** Writing – review & editing, Investigation. **Marique Aucamp:** Writing – review & editing, Resources, Investigation. **Marieta van der Rijst:** Writing – review & editing, Formal analysis. **Elizabeth Joubert:** Writing – original draft, Resources, Conceptualization.

Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Dalene de Beer reports financial support was provided by National Research Foundation. If there are other authors, they declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Abbreviations

ANOVA, analysis of variance; a_w, water activity; db, dry basis; DMSO, dimethylsulfoxide; DSA, descriptive sensory analysis; DSC, differential scanning calorimetry; FRE, fermented rooibos extract; FRE_IN, FRE spray-dried with inulin (1:1, m/m); FTIR, Fourier transform infrared; GAB, Guggenheim-Anderson-de Boer; GRE, green rooibos extract; GRE_IN, GRE spray-dried with inulin (1:1, m/m); HPLC-DAD, high-performance liquid chromatography with diode-array detection; M_0, monolayer moisture content; MSI, moisture sorption isotherm; PCA, principal component analysis; PPAG, *Z*-2-(β-D-glucopyranosyloxy)-3-phenylpropenoic acid; RE6G, (*R*)-6-β-D-glucopyranosyleriodictyol; RE8G, (*R*)-8-β-D-glucopyranosyleriodictyol; RH, relative humidity; RH_0, environmental RH where deliquescence occurs; RTD, ready-to-drink; RTR, ready-to-reconstitute; SE6G, (*S*)-6-β-D-glucopyranosyleriodictyol; SE8G, (*S*)-8-β-D-glucopyranosyleriodictyol; T_g, glass transition temperature; wb, wet basis; XRPD, X-ray powder diffraction.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jspr.2026.102954.

Data availability

Data will be made available on request.

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