



Sustainable use of coffee roasting by-products: development of high value-added gummy candies

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Abstract

The sustainable utilization of production wastes in the agri-food sector is an increasing challenge. This work aims to evaluate the applicability of coffee silverskin, the main by-product of coffee roasting, in the formulation of gummy candies. Firstly, the experimental plan envisaged the extraction and characterization of bioactive compounds from coffee silverskin. The influence of different concentrations of coffee silverskin extract (1%, 2%, and 4%) was then evaluated on chemical, physical, microbiological, structural, and sensory gummy candies characteristics for 120 storage days. Candies formulated without coffee silverskin extract were used as control. The results up to 120 days of storage revealed the higher quality of gummy candies enriched with 1%, 2% and 4% coffee silverskin extract not only for their bioactive content, ranging from 147.9 to 161.1 mg GAE Kg⁻¹ of phenolic compounds, but also for their antioxidant activity, with values at the end of storage of 15.06, 30.25, 31.50 and 28.20 μmol TE g⁻¹ respectively in control and gummy candies enriched with 1%, 2% and 4% coffee silverskin extract. Moreover, all the candies enriched with silverskin coffee extract showed better physical and sensory characteristics compared to the control taste. The results show that the proposed use of silver coffee skin improves and preserves the quality of gummy candies and then be employed as an ingredient to improve the quality of confectionery products.

Keywords Agri-food waste · Coffee roasting by-product · Coffee silverskin · Gummy candy

Introduction

Large volumes of processing wastes are generated along the food chain, not only costly to dispose of, but also producing significant damage to the surroundings when not disposed of appropriately. The reusability of processing waste in multi-hued sectors, as the food sector, has been viewed as the ideal solution to raise environmentally eco-friendly supply chains and reduce disposal expenses [1].

In recent times, coffee silverskin (CS) has been pointed out as a source of precious fibers, proteins, polyphenols, and melanoidins, suitable to ameliorate food characteristics and bring salutary benefits to the consumer [2]. In addition, coffee silverskin extracts (CSE) have been described as a safe food element in antecedent studies due to their non-carcinogenicity and low or no existence of toxic substances (ochratoxin A, pesticides, and polycyclic aromatic hydrocarbons) [3]. Potential applications of CS powders and extracts have been reported in cookies as a partial substitute for flour [4] and in chicken meat to prevent oxidation [5]. However, the influence of coffee silverskin extract on gelatinous matrices,

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as proposed in this study, has not yet been tested. In addition, due to the growing consumer claim for food products free of synthetic supplements, the development of sweet products based on natural constituents with antioxidant properties could provide a chance to realize new and healthier products for both the consumer and the confectionery industries.

The gummy candies could be considered a composite gel complex boasting gelling agents, sugars, water, and different minor constituents. This peculiar formulation makes them matrices qualified for the inclusion and retention of compounds with high added value [6].

The work aimed to test the effect of different percentages of CSE (1%, 2%, and 4%), derived by eco-friendly extraction with food-grade solvents, on healthy compounds and quality characteristics of enriched gummy candies, focusing particular attention on common defects found during the storage (crystallization of sugars, loss sensorial characteristics, presence of mold, etc.) that endanger the acceptance and safety of the product itself.

Materials and methods

Recovery of antioxidant compounds from coffee silverskin and chemical characterization

Coffee silverskin (CS) was supplied by a local coffee roaster industry (Caffè Mauro S.p.A.) and was the result of the roast of a commercial coffee blend, comprised of 50% *Coffea Arabica* and 50% *Coffea Canephora* var. Robusta beans.

After receiving, the sample was dehydrated (50 °C for 2 h) up to 10% of moisture content then it was ground, homogenized, and used to prepare extracts.

The extraction was executed by mixing 2 g of CS powder with 20 mL of hydroalcoholic solvent (EtOH 30%). The solid-liquid extractions were realized on a heating plate (60 °C) with continuity stirring for 60 min. Thereafter, the extracts were centrifuged (6000 rpm, 10 min, 20 °C) and the supernatant was recuperated, filtered using a Buchner funnel, and stored at -21 °C until further analysis. In triplicate, all determinations were carried out.

The pH and total soluble solids (TSS, ° Bx) of CS extract (CSE) were determined using a digitally calibrated pH meter (pH 4, pH 7; Crison Basic 20, Spain) equipped with an ion-selective electrode and a digital refractometer (DBR 047 SALT) respectively.

The total content of phenolic compounds (TPC) was found using the Folin-Ciocalteu colorimetric method according to Alves et al. [7] with some modifications. Briefly, 0.3 mL of a diluted CSE (1:20) was mixed with 2.5 mL of the Folin-Ciocalteu reagent (10% v/v) and 2 mL of a Na₂CO₃ solution (7.5% w/v). The mixture was incubated

for 15 min at 45 °C, and after 30 min at room temperature, absorbance readings at 765 nm were performed, against a reagent blank, using a double-beam UV spectrophotometer (Perkin-Elmer UV- Vis λ2, Waltham, Massachusetts, USA). A calibration gallic acid curve (2–10 mg L⁻¹; R² = 0.999) was used and the obtained results were expressed as mg of gallic acid equivalents (GAE) L⁻¹ of CSE.

Total flavonoid contents (TFC) were determined according to Costa et al. [8]. The reaction mixture was prepared by adding 1 mL of CSE with 4 mL of distilled water and 300 μL of NaNO₂ (25%). After 5 min at room temperature, 300 μL of 10% AlCl₃ were added, and after 1 min 2 mL NaOH (4% m/v) and 2.4 mL of ultrapure water. The absorbance at 510 nm was measured after 10 min at room temperature and total flavonoid content was calculated through a calibration epicatechin curve (0–100 mg/L; R² = 0.999) and expressed as mg of epicatechin equivalents (ECE)/ Kg of CSE.

The antioxidant activity of the extract was determined by a multitarget approach using the DPPH, ABTS, and FRAP assays. The DPPH assay was performed as described by Vimercati et al. [9] with some modifications. In brief, 40 μL of CSE (diluted 1:10) were added to 2960 μL of a 6 × 10⁻⁵ M of methanol solution of DPPH and left in darkness for 30 min at room temperature. The absorbance was assessed at 515 nm using a double-beam UV spectrophotometer (Perkin-Elmer UV- Vis λ2, Waltham, Massachusetts, USA) versus a blank (methanol).

For the ABTS assay, the methodology followed the protocol reported by Bilge et al. [10]. The FRAP assay was carried out by the method described by Benzie et al. [11] with some modifications. Briefly, 3360 μL of the FRAP reagent (consisting of 25 mL acetate buffer 0.3 M, 2.5 mL 10 mM TPTZ, and 2.5 mL 20 mM solution ferric chloride) was mixed with 40 μL of CSE. The mixture was vortexed and kept in a water bath for 6 min at 37 °C in the dark. After, the absorbance was reordered at 595 nm. The results of antioxidant assays were expressed as mM Trolox equivalents L⁻¹ of CSE, compared with a Trolox calibration curve.

The total bacterial count bacteria (TBC), yeasts, and molds were detected to evaluate the microbiological contamination of CSE by the procedure described by Nolasco et al. [12]. The sample was serially diluted and, subsequently, 1 mL of each dilution was transferred on to the surfaces of the used plates. TBC was performed by inoculating ready-to-use chromogenic plates (Compact Dry) and incubating them at 25 ± 2 °C for 48 ± 3 h. Dichloran Rose Bengal Chloramphenicol (DRBC) agar base plates were used to enumerate yeasts and molds, and the plates were incubated after solidification at 25 °C for 4–5 days before counting the colonies. The results are reported as Log₁₀ colony-forming units (CFUs) mL⁻¹ of CSE.

Table 1 Ingredients used for the formulation of gummy candies

Ingredients (g/ 100 g of gummy candies)	Sample			
	CTR	CS1	CS2	CS3
Sucrose	31	31	31	31
Glucose syrup	28	28	28	28
Pork gelatin	8	8	8	8
Apricot juice	22	22	22	22
Citric acid	1	1	1	1
Extract coffee silverskin	-	1	2	4
Water	10	9	8	6

Gummy candy manufacturing

The gummy candies were made as described by Miranda et al. [13] with some modifications, the ingredients are reported in Table 1. A mixture of apricot juice, sucrose, glucose syrup (40 DE), and citric acid was heated at 85 °C under stirring for complete dissolution. After cooling to approximately 50 °C, CSE was added and homogenized for 10 min. The pork gelatin sheets (240 °Bloom) were then added to water for 10 min, to favor the hydration, and finally, the gelatin was added to the mixture and homogenized at 50 °C ± 5 °C under stirring for complete dissolution. The jelly mass was immediately placed in silicone molds and dried in an aseptic environment by using a vertical laminar flow hood (UV lamp 30 W, mod. ASALAIR 1200 FLV, Asal Srl, Milan, Italy) for 72 h at 25 °C until the candies reached the water activity (a_w) less than 0.70.

Subsequently, the gummy candies were demolded, packed in waxed paper, and stored in darkness in constant climate chambers (25 °C) for 120 days. Gummy candies samples were identified as follows: CTR (0% CSE), CS1 (1% CSE), CS2 (2% CSE), and CS3 (4% CSE) (Fig. 1).

Fig. 1 Gummy candies after formulation. (CTR: gummy candies without CSE; CS1: gummy candies with 1% CSE; CS2: gummy candies with 2% CSE; CS3: gummy candies with 4% CSE)



Physicochemical and microbial analyses of gummy candies

Physicochemical analyses were performed immediately after candy production (0 days) and, subsequently, after 15, 30, 60, 90, and 120 days of storage. The sensory and microbiological analyses were carried out immediately after the formulation and after 120 days.

Color

Color analysis was evaluated using a Spectrophotometer using a D65 illuminant (Minolta CM-700 d, Japan), with measurement on twenty-four points for each sample.

The results of the colorimetric measurements were expressed in the CIE L*a*b* scale adopted as standard by the International Commission on Illumination. The total color difference (ΔE) after 120 days of storage was obtained by using the following Eq. (1):

$$\Delta E = [(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2]^{1/2} \quad (1)$$

Water activity, pH, total soluble solids, moisture content, and microbial analyses

The water activity (a_w), pH, and total soluble solids (TSS) were measured according to Cedeño-Pinos et al. [14]. The moisture content was carried out using the gravimetric technique, according to the Association of Official Analytical Collaboration [15].

The mold enumeration was performed on Dichloran Rose Bengal Chloramphenicol (DRBC) base plates. Briefly, 10 g of each sample was placed in a sterile bag with 100 ml of Ringer solutions, homogenated with Stomacher (Bag-Mixer® 400 P, Interscience, France) for 3 min. The resulting

microbiological suspension was diluted in series then, 1 ml of each dilution was placed on the plates. The enumeration of molds was made after incubation at 25 °C for 4–5 days. The results were expressed as \log_{10} CFU g^{-1} of gummy candies.

Total phenolics content and antioxidant activity

The samples (6 g) were dissolved with 20 ml methanol under agitation for 45 min at room temperature, to estimate the total content of phenolic compounds and the antioxidant activity of gummy candies. The mixture was then centrifuged (8000 rpm and 4 °C for 10 min), the supernatant was recovered, filtered (PTFE 0.45 μ m, diameter 15 mm), and frozen at -80 °C until analysis.

The total content of phenolic compounds (TPC) in gummy candies was determined using the Folin–Ciocalteu with an experimental procedure proposed by Cedeño-Pinos et al. [14] with modifications. In a 10 ml graduated flask were mixed 1 ml of CSE, 5 ml of distilled water, and 0.8 ml of Folin–Ciocalteu reagent were mixed. After 8 min at room temperature and under constant stirring, 1.2 ml of 20% (v/v) Na_2CO_3 was added. Then the reaction mixture was completed to a volume of 10 ml with distilled water and incubated in a dark at room temperature for 2 h. The absorbance was obtained at 760 nm using a spectrophotometer (Perkin-Elmer UV–Vis k2, Waltham, Massachusetts, U.S.). A mixture reaction without a sample was used as a blank. The results were expressed as μ g gallic acid equivalents (GAE) g^{-1} of gummy candies using a calibration curve of gallic acid as a standard ($R^2 = 0,9996$).

Literature shows that the ABTS test is the best way to test the antioxidant activity of gummy candies [16]. Then, the antioxidant activity of gummy candies was tested with ABTS assay considering both the matrix of the tested food and the antioxidant activity performed by the compounds present in CSE to extinguish the $ABTS^+$ in lipophilic and hydrophilic environments by the procedure described by Re et al. [17] with some modifications. ABTS radical cation was generated by mixing the 7 mM ABTS stock solution with 2.45 mM potassium persulfate and allowing the mixture to stay in the dark at room temperature for 12–16 h before use. Then, it was balanced to 30 °C using PBS 0.1 M (pH 7.4) up to an absorbance of 0.70 ± 0.02 to 734 nm. For sample analysis, 100 μ L of sample extract and 2900 μ L ABTS were mixed and incubated in darkness for 6 min. Sample absorbance was measured at 734 nm against a blank (PBS). A calibration curve was prepared with Trolox, and the results were expressed as μ M Trolox Equivalents (TE) g^{-1} of gummy candies.

Textural properties of gummy candies

To determine the influence of CSE on the structure of gummy candies various parameters were monitored periodically at time 0, 15, 30, 60, 90, and 120 days of storage.

The texture was evaluated with two types of tests: texture profile analysis (TPA) and the perforation test following the methods described by Teixeira-Lemos et al. [18].

All tests were conducted by measuring force on compression, using a 50 kg load cell and a trigger force of 0.05 N. All mechanical properties were measured on 7 jellies gummies of each type, at room temperature.

The texture profile analysis (TPA) was conducted with a flat cylindrical probe P/50, the compression distance was 5 mm, and the pre-test and post-test speeds were all equal to 0.5 mm/s. Two compression cycles were performed with a 5 s interval between them. The texture profile analysis on samples was expressed in terms of: hardness (N) as the force required to compress the sample; springiness (mm) as thickness recovered from the sample between the first and second compression; cohesiveness (no units) as the force due to the interaction among the various ingredients used for the formulation of the sample, and expressed as the ratio between the areas of the curves (force x time) obtained during the first and second compression; chewiness (N) as energy required to chew food before swallowing, and expressed as the hardness x cohesiveness x springiness; stickiness (N) expressed as the force necessary to resist the separation of two surfaces in contact (caramel and probe).

The perforation tests with a probe P/5 with cylindrical termination with a 5 mm diameter. This test utilized a perforation distance of 3 mm, pre-test speed of 2.0 mm/s, test speed of 1.0 mm/s, and post-test speed of 1.0 mm/s.

Quantitative descriptive sensory analysis of gummy candies

A sensory quantitative sensory descriptive analysis (QDA) was performed by recruiting ten panelists (five men and five women aged between 22 and 45 years) among departmental students and faculty staff of the Mediterranean University of Reggio Calabria with previous experience in sensory analyses. The participants were trained before the sessions to identify, select and quantify the main sensory descriptors for gummy candies [19]. Table 2 shows the olfactory and taste descriptors, and the reference used to train the participants. Visual appearance (brilliance, intensity of yellow, intensity of orange, intensity of brown) and texture descriptors (consistency, softness, gumminess, adhesiveness) were identified, selected and quantified based on the previous experience of the panelist. The quantification of sensorial attributes was carried out using a hedonic scale, from 0 to 10

Table 2 Olfactory and taste descriptors and corresponding references used to train the panelist

Descriptor	References
<i>Taste</i>	
Sweet	Sucrose solution (0–10 g L ⁻¹)
Fruity	Apricot juice solution (0–1000 mL L ⁻¹)
Citrus	Lemon juice solution (0–1000 mL L ⁻¹)
Astringent	Acid tannic solution (0–0.1 g L ⁻¹)
Aftertaste	Time of persistence of taste sensations after swallowing
<i>Olfactory</i>	
Fruity	Apricot aroma (0–20 drops)
Citrus	Lemon aroma (0–20 drops)
Intensity	Intensity perceived by the combination of fruity and citrus descriptors

The reference taste solutions were made with distilled water. The reference of aromas was placed on strips for aromas

points, where a score of 0 indicates the absence of the attribute, 10 indicates the total presence of the attribute and 5 score was fixed as the minimum acceptable (Fig. 2). In addition, the panelists were asked to give an overall score from 0 to 10 based on the overall judgment of the taste, olfactory, visual and texture attributes considered (total acceptability). Sensory analysis of the gummy candies was conducted in opportune tasting booths (90 cm), equipped with sinks, lighting and shelves for samples. Participants were given water to rinse their palate during the sensory session.

The sensory analysis was carried out on gummy candies samples at the beginning (1 day) and end of storage (120 days). The results were reported as an average of the evaluations.

Statistical analysis

The analytical data were reported as means \pm standard deviations of replicates. The analysis of variance (one-way ANOVA) was conducted by applying Tukey's *post-hoc* at

Table 3 pH, total soluble solids (TSS), total phenolics content (TPC), total flavonoid content (TFC) and antioxidant activity (DPPH, ABTS and FRAP assay) of coffee silverskin extract

Parameter	Results
pH	8.53 \pm 0.00
TSS ($^{\circ}$ Bx)	9.30 \pm 0.00
TPC (mg GAE L ⁻¹)	1954.75 \pm 3.13
TFC (mg ECE L ⁻¹)	1426.21 \pm 67.4
DPPH (mmol Trolox L ⁻¹)	207.08 \pm 9.95
ABTS (mmol Trolox L ⁻¹)	330.16 \pm 6.30
FRAP (mmol Trolox L ⁻¹)	57.78 \pm 7.80

$p < 0.05$ by Version 20.0 SPSS software (SPSS Inc., Chicago, IL, USA).

Results and discussion

Chemical characterization of coffee silverskin extract

The TSS and pH values of coffee silverskin extract (CSE) were 8.53 and 9.3, respectively.

The spectrophotometric analysis showed a high content in TPC (1954.75 \pm 3.13 mg GAE L⁻¹), TFC (1426.21 \pm 67.4 mg ECE L⁻¹) and strong antioxidant activity (ABTS 330.16 \pm 6.3 mmol Trolox L⁻¹, DPPH 207.08 \pm 9.95 mmol Trolox L⁻¹ and FRAP 57.78 \pm 7.8 mmol Trolox L⁻¹) of CSE (Table 3).

The obtained results agree with previous studies [20, 21] and are closely related to the variables used during the extraction process. Indeed, it has been observed that the extractability of CS antioxidant compounds is affected by the polarity of the used solvent and may be related to the fact that many phenolic compounds often have intermediate solubility. However, it should be noted that the chemical profile of CS can be strongly influenced by different variables,

Name and surname _____ Date _____ Sample n° _____

The judge assesses the organoleptic characteristics of the product according to the following scale of intensity

VISUAL APPEARANCE											
	0	1	2	3	4	5	6	7	8	9	10
Brilliance											
Intensity of yellow											
Intensity of orange											
Intensity of brown											

OLFACTORY SENSATIONS											
	0	1	2	3	4	5	6	7	8	9	10
Intensity											
Fruity											
Citrus											

TEXTURE											
	0	1	2	3	4	5	6	7	8	9	10
Consistency											
Softness											
Gumminess											
Adhesiveness											

TASTE											
	0	1	2	3	4	5	6	7	8	9	10
Sweet											
Fruity											
Citrus											
Astringent											
Aftertaste											

TOTAL ACCEPTABILITY											
	0	1	2	3	4	5	6	7	8	9	10

Fig. 2 Gummy candies sensory descriptors list

including coffee varieties, production environment, climatic conditions, field treatments, methods of coffee processing, and methods of storage of the by-product [22].

Finally, the microbiological analysis did not provide evidence of the microbial presence in extracts: it is plausible that the high temperature of the roasting process and the low moisture content limited its microbial load and extended its storage [23, 24].

Physico-chemical and microbiological characterization of gummy candies

Table 4 shows the results of physicochemical analyses of gummy candy samples immediately after the preparation (time 0) and after 120 days of storage at 25 °C.

Colour

Colour is an important quality index for all foods and is linked to the overall acceptability of products by the final consumer. Table 4 shows the results relating to the colorimetric measurement of the different formulations of gummy candies, immediately after the preparation (time 0) up to 120 days of storage at 25 °C. The gelling process, shelf-life, and physical-chemical properties of gummy candies could be influenced by the applied extracts, as described by Delgado et al. [25]. Difference in L*, a*, and b* values was observed among CTR and CS1, CS2 and CS3. The addition of CSE has predictably led to a progressive browning (decrease of L*) of the candies and a visible variation of colour from bright orange to orange-brown (decrease of a* and b*). This trend was maintained throughout storage. The

significant found colour differences were attributable to the presence in CSE of melanoidins, responsible for the brown colour of many foods [26]. Moreover, the liquid nature of the CSE allowed a better dispersion in the gelatinous matrix of candies and, consequently, the strong variation of colour in all enriched candies than no enriched ones [27].

A significant increase in the L* value was observed in CTR after 60 days of storage, probably due to the crystallization of the sugar that occurred following of moisture adsorption and desorption from the surroundings [28]. It was reported already that during the storage of gummy candies, a slow migration of moisture could happen inside the package, causing the lowering of the glass transition temperature, greater mobility of the molecules embedded in the matrix, and, as a result, the crystallization of the sucrose, loss of the aromatic characteristics, hardening and a change in the colour of the candies [29, 30].

The advantageous impact of CS on the support of colour was too affirmed by the results of overall colour change (ΔE) ranging from 1.65 to 3.61, where the highest value recorded in CTR and the lowest one in CS2 after 120 days of storage. The obtained results suggest that the presence of 2% and 4% CSE in the formulation does not affect the visual appearance and, consequently, the visual acceptability of the product for 120 days of storage.

Water activity, moisture content, PH, total soluble solids and mold count of gummy candies

The gummy candies are characterized by a colloidal system containing gelling agents, sugars, water, and other minor constituents. For this reason, these foods are highly

Table 4 Colour parameter of gummy candies during the storage period

Parameter	Sample	Storage time (days)					Sig.	
		0	15	30	60	90		120
Lightness (L*)	CTR	49.2 ^{aAB}	50.6 ^{aA}	49.1 ^{aAB}	48.7 ^{aAB}	48.2 ^{aB}	50.5 ^{aA}	**
CIE units	CS 1	47.4 ^{bAB}	48.3 ^{abA}	47.7 ^{abAB}	48.3 ^{aA}	46.6 ^{aB}	47.9 ^{bAB}	*
	CS 2	45.9 ^{cA}	44.9 ^{bA}	46.4 ^{bA}	46.0 ^{bA}	46.1 ^{aA}	46.3 ^{bA}	ns
	CS 3	46.6 ^{bA}	46.1 ^{bA}	47.2 ^{bA}	45.4 ^{bA}	48.3 ^{aA}	46.6 ^{bA}	ns
Sig.		**	**	**	**	ns	**	
Redness (a*)	CTR	2.04 ^{bBC}	2.77 ^{aA}	2.34 ^{aABC}	1.78 ^{aC}	2.41 ^{aAB}	2.43 ^{aAB}	**
CIE units	CS 1	2.49 ^{aA}	2.65 ^{aA}	2.84 ^{aA}	1.44 ^{bB}	1.44 ^{bB}	1.73 ^{bB}	**
	CS 2	1.71 ^{bA}	1.42 ^{bAB}	1.21 ^{bAB}	1.02 ^{cB}	1.01 ^{cB}	1.51 ^{bA}	**
	CS 3	0.87 ^{cAB}	0.99 ^{bA}	0.81 ^{bAB}	0.59 ^{dB}	0.75 ^{cAB}	0.71 ^{cAB}	**
Sig.		**	**	**	**	**	**	
Yellowness (b*)	CTR	6.49 ^{aA}	6.67 ^{aA}	6.32 ^{aA}	5.07 ^{aB}	5.85 ^{aAB}	4.99 ^{aB}	**
CIE units	CS 1	5.58 ^{bAB}	5.54 ^{bABC}	6.06 ^{aB}	4.90 ^{aBCD}	4.47 ^{bCD}	3.86 ^{bD}	**
	CS 2	3.71 ^{cA}	3.84 ^{cA}	3.71 ^{bA}	3.40 ^{bAB}	3.79 ^{cA}	2.82 ^{cB}	**
	CS 3	3.04 ^{cBC}	3.88 ^{cA}	3.64 ^{bAB}	2.69 ^{cCD}	3.55 ^{cAB}	2.35 ^{cD}	**
Sig.		**	**	**	**	**	**	

Small letters within a column and capital letters within a row show significant differences as assessed by Tukey's *post hoc* test. Abbreviations: **, significance at $p < 0.01$; *, significance at $p < 0.05$; n.s., not significant. CTR: gummy candy without CSE; CS1: gummy candy with 1% CSE; CS2: gummy candy with 2% CSE; CS3: gummy candy with 4% CSE

hygroscopic and present serious difficulties in drying, stabilization, and storage [31]. Typically, all gummy candies are affected by a progressive loss of moisture during storage that can be attributed to many factors, such as formulation ingredients, storage temperature, and packaging materials [25]. In addition, due to the hygroscopic characteristics, continuous migration of moisture between the candy and the surrounding environment may cause chemical-physical, sensory, and microbiological variations. In gummy candies, the ideal moisture content value to allow long storage and preserve the physicochemical characteristics over time should be between 8% and 22% [28].

In this study, enrichment with liquid CSE influenced the moisture content as well as the stabilization time of all treated candies. In fact, in the first storage period (up to 15 days) a faster water loss in CS1, CS2, and CS3 was observed compared to CTRL. Probably, the interaction between the basic ingredients (used to formulate candies) and CS determined a change of structure in gummy candies and, consequentially, the delay of stabilization. A low and smooth significant decrease of moisture occurred from 15 to 120 days. The great loss of moisture recorded in the

enriched candies was due to the hydroalcoholic nature of the extract and the progressive increase of its concentration in the candy formulation. Matulyte and colleagues [32] found that the use of ethanolic extracts in gummy candies formulation affected the moisture loss more than other samples because alcoholic and hydroalcoholic extracts evaporate faster than other ones.

Water activity (a_w) is essential to maintain the chemical and storage stability of food products and is closely related to food microbiological safety. As shown in Table 5, differences among enriched (CS1, CS2, and CS3) and not enriched (CTRL) candies were found throughout the whole storage period also for this parameter. A_w was affected by the hygroscopicity and intermolecular interactions encouraged by the presence of additional solutes in of CS1, CS2, and CS3. The a_w values of all gummy candies decreased from 0.66 to 0.58 in CTRL, from 0.66 to 0.55 in CS1, and from 0.65 to 0.64 in CS2 up to the end of storage. In CS3 the values remained constant. The a_w results found in this study ensure the safety and physicochemical quality of the final product and agree with previous studies where the value of

Table 5 Moisture content, water activity, pH, total soluble solids and mold of gummy candies

Parameter	Sample	Storage time (days)						Sig.
		0	15	30	60	90	120	
Moisture (g/100 g)	CTRL	19.87 ^{aA}	19.41 ^{aA}	19.61 ^{aA}	19.80 ^{aA}	16.62 ^{bB}	17.80 ^{aB}	**
	CS 1	19.97 ^{aA}	18.30 ^{bBC}	18.75 ^{aB}	17.51 ^{bC}	20.21 ^{aA}	16.82 ^{bD}	**
	CS 2	18.50 ^{bA}	16.10 ^{cB}	16.52 ^{bB}	15.40 ^{dC}	14.95 ^{cC}	16.53 ^{bB}	**
	CS 3	18.30 ^{bA}	15.90 ^{cBC}	15.37 ^{bC}	16.13 ^{cB}	13.63 ^{dD}	13.52 ^{cD}	**
Sign		**	**	**	**	**	**	
Water activity (a_w)	CTRL	0.65 ^{bC}	0.68 ^{aA}	0.67 ^{cB}	0.67 ^{aB}	0.61 ^{bD}	0.58 ^{cE}	**
	CS 1	0.66 ^{aB}	0.67 ^{bA}	0.67 ^{cA}	0.67 ^{aA}	0.59 ^{dC}	0.55 ^{dD}	**
	CS 2	0.64 ^{cC}	0.67 ^{bB}	0.68 ^{bA}	0.67 ^{aB}	0.63 ^{aD}	0.64 ^{aC}	**
	CS 3	0.60 ^{dC}	0.66 ^{bB}	0.69 ^{aA}	0.66 ^{bB}	0.60 ^{cC}	0.60 ^{bC}	**
Sign		**	**	**	**	**	**	
TSS (° Bx)	CTRL	49.91 ^{cF}	56.15 ^{cD}	56.91 ^{bC}	53.33 ^{dE}	59.72 ^{cB}	60.61 ^{dA}	**
	CS 1	50.36 ^{bD}	58.72 ^{bB}	55.00 ^{dC}	58.20 ^{cB}	55.10 ^{dC}	65.10 ^{aD}	**
	CS 2	53.56 ^{aD}	61.35 ^{aB}	57.20 ^{bC}	63.32 ^{aA}	60.91 ^{bB}	61.00 ^{cA}	**
	CS 3	50.42 ^{bF}	63.61 ^{aB}	61.51 ^{aC}	60.01 ^{bE}	65.14 ^{aA}	61.00 ^{bD}	**
Sign		**	**	**	**	**	**	
pH	CTRL	3.86 ^{bC}	3.84 ^{cC}	3.86 ^{dC}	3.91 ^{cB}	3.96 ^{dA}	3.90 ^{cB}	**
	CS 1	3.85 ^{bD}	3.89 ^{bC}	3.91 ^{cC}	3.95 ^{bB}	3.99 ^{cA}	3.90 ^{cB}	**
	CS 2	3.93 ^{aD}	3.91 ^{aE}	3.96 ^{bC}	3.97 ^{bC}	4.05 ^{bA}	4.00 ^{bB}	**
	CS 3	3.94 ^{aD}	3.92 ^{aE}	4.00 ^{aBC}	4.01 ^{aB}	4.08 ^{aA}	3.99 ^{aC}	**
Sign		**	**	**	**	**	**	
Molds (Log UFC/g)	CTRL	n.d.	-	-	-	-	n.d.	
	CS 1	n.d.	-	-	-	-	n.d.	
	CS 2	n.d.	-	-	-	-	n.d.	
	CS 3	n.d.	-	-	-	-	n.d.	

Small letters within a column and capital letters within a row show significant differences as assessed by Tukey's *post hoc* test. Abbreviations: **, significance at $p < 0.01$; *, significance at $p < 0.05$; n.s., not significant. CTRL: gummy candies without CSE; CS1: gummy candies with 1% CSE; CS2: gummy candies with 2% CSE; CS3: gummy candies with 4% CSE

a_w for jellies and candies should be between 0.55 and 0.75 [33].

Contrary to the trend shown of moisture and a_w , total soluble solids increased significantly during the storage: this is related to both progressive dehydration of food and to chemical changes at the expense of sugars present in the complex matrix of gummy candies with a progressive polysaccharides' conversion into monosaccharides and oligosaccharides during their shelf-life, confirmed by literature [34].

All quality parameters' observed trends (a_w , moisture, and total soluble solids) suggested the need for more barricaded packaging to better protect the candies from dehydration and, consequently, from all the related changes over time [28].

Moreover, significant differences in pH were found throughout the storage time among the candies: greater in CS1, CS2, and CS3 than in CTRL and caused by the original characteristics of the used extract (pH=8.9).

The formulation of gummy candies has ensured food safety by avoiding the proliferation of mold during storage (data not shown): it is due to the maintenance of a_w , pH, and moisture values below the threshold limits, as reported in previous studies [18, 32].

Total phenolic content and antioxidant activity

The addition of CSE influenced significantly ($p < 0.05$) also the total phenolic content in gummy candies, closely linked to the extract's concentration used in the formulation (Table 6).

Immediately after preparation, the phenolic compounds in all CS gummy candies were significantly higher than CTRL (265.07 $\mu\text{g GAE g}^{-1}$, 272.70 $\mu\text{g GAE g}^{-1}$, 282.31 $\mu\text{g GAE g}^{-1}$, 317.09 $\mu\text{g GAE g}^{-1}$ for CTRL, CS1, CS2 and CS3, respectively) and this trend was maintained throughout their

storage. However, all candies, enriched or not, showed a significant ($p \leq 0.05$) decrease in phenolic compounds during storage. This trend may have been caused by storage conditions (packaging and temperature), both the presence of oxygen and the temperature (25 °C) could have advantaged the natural degradation and the trigger of chemical reactions, causing of decrease in these precious compounds. Moreover, the change of moisture observed during storage has facilitated the mobilization of phenolic compounds present in the matrix and increased their susceptibility to degradation [28]. Nevertheless, the values of phenolic compounds found in candies enriched with CSE in this study were significantly higher than those of other potentially functional candies formulated with banana and Malaysian sting-less bee honey (183 $\mu\text{g GAE g}^{-1}$) [35] and comparable with those obtained in previous studies using phenolic extract of liquid Rosemary Extract (190–273 $\mu\text{g GAE g}^{-1}$) [14], peppermint (160–380 $\mu\text{g GAE g}^{-1}$) [36], and Gummy Candies Made with sugars/fructans and green Propolis (153–271 $\mu\text{g GAE g}^{-1}$) [37].

Regarding the antioxidant activity, the values show significant differences ($p < 0.05$) among all gummy candies tested (Table 6). The antioxidant activity was dose-dependent since a significant increase was encountered after the addition of crescent percentages of CSE. The distinction between enriched and not enriched candies was evident immediately after the formulation and this tendency was maintained for the whole conservation, with ranges from 24.44 to 15.06 $\mu\text{mol TE g}^{-1}$ for CTRL, 38.65–30.25 $\mu\text{mol TE g}^{-1}$ for CS1, 42.81–31.50 $\mu\text{mol TE g}^{-1}$ for CS2 and 45.75–28.20 $\mu\text{mol TE g}^{-1}$ for CS3.

Comparing the results between TPC and antioxidant activity, it was evident the radical scavenging activity was influenced not only by phenolic compounds but also by other compounds present in the CSE. While showing an

Table 6 Total phenolic content (TPC) and antioxidant activity (ABTS assay) of gummy candies during storage period

Parameter	Sample	Storage time (days)						Sig.
		0	15	30	60	90	120	
TPC ($\mu\text{g GAE g}^{-1}$)	CTRL	265.11 ^{cA}	235.40 ^{cB}	240.01 ^{bB}	188.86 ^{bC}	179.91 ^{bC}	131.40 ^{bcD}	**
	CS 1	272.71 ^{bcA}	262.31 ^{bAB}	246.80 ^{bb}	205.82 ^{bC}	207.65 ^{bC}	161.12 ^{aD}	**
	CS 2	282.38 ^{ba}	256.89 ^{bb}	241.60 ^{bC}	191.24 ^{bD}	189.52 ^{aD}	128.00 ^{cE}	**
	CS 3	317.15 ^{aA}	295.10 ^{aAB}	278.91 ^{aB}	240.96 ^{aC}	224.06 ^{aC}	147.90 ^{abD}	**
Sign		**	**	**	**	**	**	
ABTS ($\mu\text{mol TE g}^{-1}$)	CTRL	24.43 ^{cA}	18.41 ^{cBC}	17.07 ^{cBC}	14.63 ^{bCD}	19.68 ^{bB}	15.06 ^{bD}	**
	CS 1	38.65 ^{ba}	36.61 ^{bAB}	33.73 ^{bCD}	29.59 ^{aC}	33.99 ^{aAB}	30.25 ^{aC}	**
	CS 2	42.81 ^{aA}	37.61 ^{abB}	37.80 ^{aB}	29.83 ^{aC}	35.78 ^{aB}	31.49 ^{aC}	**
	CS 3	45.75 ^{aA}	41.20 ^{aB}	38.98 ^{aB}	31.86 ^{aCD}	34.13 ^{aC}	28.20 ^{aD}	**
Sig.		**	**	**	**	**	**	

Small letters within a column and capital letters within a row show significant differences as assessed by Tukey's post hoc test. Abbreviations: **, significance at $p < 0.01$; *, significance at $p < 0.05$; n.s., not significant. CTRL: gummy candy without CSE; CS1: gummy candy with 1% CSE; CS2: gummy candy with 2% CSE; CS3: gummy candy with 4% CSE

important decrease in phenolic compounds, the antioxidant activity of enriched candies remained high until the end of storage. Probably, this can be explained by the simultaneous action of various compounds among which the most important were chlorogenic acid and melanoidins [21, 38]. The presence of these compounds explains the difference between the values of total phenolic compounds and the antioxidant activity of CS1, CS2, and CS3 from the beginning to the end shelf life of gummy candies. The results of antioxidants quantified in CS1, CS2, and CS3 were similar and greater to those observed in candies formulated with other ingredients [16, 39–41].

Textural properties of gummy candies

The textural changes observed during storage of the gummy candies are shown in Table 7. The obtained results indicated that the addition of CSE significantly influenced the

hardness, chewiness, stickiness of all treated samples. Nevertheless, it was equally evident that the factor that most affected the maintenance of structural characteristics during the shelf-life of gummy candies was the time and condition of storage.

As expected, all chemical variations had a significant effect on all the analysed textural variables. Certainly, a fundamental role in hardness, cohesiveness, chewiness and stickiness was played by the loss of moisture. Previously, it was reported that the change of moisture over time caused a rapid increase in the viscosity of the surface area (higher stickiness) and an increase in the glass transition temperature of the candies [42, 43]. This last phenomenon determines a greater mobilization of the compounds and, subsequently, the crystallization of the sugars present in the gummy matrix and an excessive hardening of the candy [19, 43]. The dynamics described above occurred after the 60th day of storage and resulted in significant structural change

Table 7 Textural proprieties of gummy candies during the storage period

Parameter	Sample	Storage time (days)						Sig.
		0	15	30	60	90	120	
Hardness (N)	CTRL	1.75 ^{abE}	3.01 ^{aD}	3.02 ^{abD}	6.17 ^{aC}	19.22 ^{aA}	13.71 ^{abB}	**
	CS 1	1.47 ^{bE}	2.45 ^{bDE}	2.66 ^{bD}	5.59 ^{aC}	13.74 ^{cA}	11.01 ^{bB}	**
	CS 2	1.81 ^{aC}	2.82 ^{abC}	2.97 ^{abC}	6.06 ^{ab}	17.46 ^{bA}	18.10 ^{aA}	**
	CS 3	1.60 ^{abE}	2.89 ^{abDE}	3.16 ^{aD}	6.66 ^{aC}	17.59 ^{bA}	14.06 ^{abB}	**
Sign		*	*	*	ns	**	*	
Springiness (mm)	CTRL	0.95 ^{bA}	0.92 ^{aA}	0.93 ^{aA}	0.98 ^{aAB}	0.84 ^{aB}	0.88 ^{aAB}	**
	CS 1	0.93 ^{abA}	0.94 ^{aA}	0.92 ^{aA}	0.91 ^{aAB}	0.84 ^{aB}	0.86 ^{aAB}	*
	CS 2	0.97 ^{aA}	0.95 ^{aA}	0.94 ^{aAB}	0.89 ^{aB}	0.89 ^{aB}	0.9 ^{aBC}	**
	CS 3	0.97 ^{aA}	0.93 ^{aABC}	0.93 ^{aAB}	0.92 ^{aABC}	0.89 ^{aBC}	0.87 ^{aC}	*
Sign.		*	ns	ns	ns	ns	ns	
Cohesiveness (No unit)	CTRL	0.95 ^{aA}	0.95 ^{aA}	0.94 ^{aAB}	0.91 ^{aBC}	0.89 ^{aC}	0.88 ^{bC}	**
	CS 1	0.96 ^{aA}	0.95 ^{aA}	0.95 ^{aAB}	0.91 ^{aBC}	0.90 ^{aC}	0.89 ^{aC}	**
	CS 2	0.96 ^{aA}	0.95 ^{aA}	0.95 ^{aA}	0.91 ^{aB}	0.91 ^{aB}	0.91 ^{abB}	**
	CS 3	0.96 ^{aA}	0.95 ^{aA}	0.94 ^{aA}	0.91 ^{aB}	0.87 ^{aC}	0.88 ^{bC}	**
Sign.		ns	ns	ns	ns	ns	*	
Chewiness (N x mm)	CTRL	9.02 ^{cC}	17.56 ^{bC}	16.36 ^{bC}	33.22 ^{bB}	58.59 ^{aA}	54.11 ^{aA}	**
	CS 1	8.99 ^{cD}	23.27 ^{aCD}	17.13 ^{bD}	33.65 ^{bC}	74.40 ^{aA}	54.65 ^{aB}	**
	CS 2	12.65 ^{aD}	23.27 ^{aD}	21.34 ^{abD}	35.25 ^{bC}	73.12 ^{aA}	61.40 ^{aB}	**
	CS 3	10.80 ^{bD}	24.67 ^{aC}	24.57 ^{aC}	47.35 ^{aB}	59.23 ^{aA}	66.24 ^{aA}	**
Sign.		**	**	*	**	ns	ns	
Stickiness (N)	CTRL	-0.05 ^{aA}	-0.21 ^{aA}	-0.41 ^{aAB}	-0.82 ^{abABC}	-1.60 ^{aD}	-1.05 ^{abCD}	**
	CS 1	-0.15 ^{bA}	-0.41 ^{aAB}	-0.69 ^{aABC}	-1.06 ^{bCDE}	-1.40 ^{aDE}	-1.53 ^{bE}	**
	CS 2	-0.10 ^{abA}	-0.43 ^{aAB}	-0.76 ^{aAB}	-1.03 ^{abB}	-1.04 ^{aB}	-0.38 ^{aAB}	*
	CS 3	-0.05 ^{aA}	-0.19 ^{aA}	-0.41 ^{aA}	-0.54 ^{aAB}	-1.47 ^{aB}	-0.55 ^{abAB}	**
Sign.		*	ns	*	*	ns	*	

Small letters within a column and capital letters within a row show significant differences as assessed by Tukey's *post hoc* test. Abbreviations: **, significance at $p < 0.01$; *, significance at $p < 0.05$; n.s., not significant. CTRL: gummy candies without CSE; CS1: gummy candies with 1% CSE; CS2: gummy candies with 2% CSE; CS3: gummy candies with 4% CSE

of all samples. However, the enriched candies maintained over time better structural characteristics in terms of stickiness, springiness, and hardness than no enriched samples. This was probably due to the nature of the binding of water to the components present in the extract, the presence of fibers could be the cause of the maintenance of consistency [12, 13].

Quantitative descriptive sensory analysis of gummy candies

The results of the quantitative descriptive sensory analysis of gummy candies is illustrated in Tables 8, 9, 10 and 11.

In particular, the visual appearance shows significant variation of yellow and brown intensity related to the different formulations and the storage times: it was probably due to the presence of melanoidins in CSE [26] and, consequently, its concentration in enriched gummy candies.

The olfactory sensations tended generally to increase during the storage in all the candy samples without significant differences, probably due to the change in a_w values, as reported by Ergun et al. [27].

The quantitative descriptive sensory analysis of gummy candies showed softness and gumminess reduction without differences after 120 days of storage for all samples. Regarding the taste sensations, CS2 candies stood out at the end of preservation for the highest scores for sweet and fruity tastes. Aftertaste tended to increase in all candies during the storage with fewer variations in CS-enriched ones than in CTR.

The results of total acceptability of the CS2 candies were higher at both storage times (7.66 at time 0 and 7.33 after 120 days) than the other samples (Fig. 3).

Table 8 Visual appearance of gummy candies at the beginning (1 day) and the end (120days) storage period

SAMPLE	Brilliance		Intensity of yellow		Intensity of orange		Intensity of brown		Sig.
	1 day	120 days	1 day	120 days	1 day	120 days	1 day	120 days	
CTR	5.8	5.2 ^{ab}	7.3 ^a	6.3 ^a	4.3	3.2 ^b	1.5 ^a	2.0 ^a	n.s.
CS1	6.7	5.8 ^a	6.2 ^a	4.2 ^b	5.2	5.5 ^a	2.7 ^a	4.3 ^b	*
CS2	4.8	4.8 ^{ab}	2.2 ^b	3.7 ^b	4.8	4.5 ^{ab}	6 ^b	4.8 ^b	n.s.
CS3	4.7	3.5 ^b	2.5 ^b	2.7 ^b	3.8	4.5 ^{ab}	8.2 ^a	7 ^a	*
Sign.	n.s.	*	**	**	n.s.	*	**	**	**

Small letters within a column show significant differences as assessed by Tukey's *post-hoc* test. Abbreviations: **, significance at $p < 0.01$; *, significance at $p < 0.05$; n.s., not significant. CTR: gummy candies without CSE; CS1: gummy candies with 1% CSE; CS2: gummy candies with 2% CSE; CS3: gummy candies with 4% CSE

Table 9 Olfactory sensation of gummy candies at the beginning (1 day) and the end (120days) storage period

SAMPLE	Intensity			Fruity			Citrus		
	1 day	120 days	Sign.	1 day	120 days	Sign.	1 day	120 days	Sign.
	CTR	1.5	4.3	**	1.6	5.7	**	1.0	4.0
CS1	2.2	5.2	**	1.8	4.8	**	1.8	4.2	*
CS2	1.7	4.5	**	2.0	5.3	**	1.8	3.8	**
CS3	2.5	4.2	*	2.7	4.5	*	1.7	2.8	n.s.
Sign.	n.s.	n.s.		n.s.	n.s.		n.s.	n.s.	

Abbreviations **: significance at $p < 0.01$; *, significance at $p < 0.05$; n.s., not significant. CTR: gummy candies without CSE; CS1: gummy candies with 1% CSE; CS2: gummy candies with 2% CSE; CS3: gummy candies with 4% CSE

Table 10 Texture of gummy candies at the beginning (1 day) and the end (120days) storage period

SAMPLE	Consistency			Softness			Gumminess			Adhesiveness		
	1 day	120 days	Sign.	1 day	120 days	Sign.	1 day	120 days	Sign.	1 day	120 days	Sign.
	CTR	7.0	5.5 ^{ab}	*	5.3	3.5	*	7.0	5.3	**	4.3	4.8
CS1	6.5	4.5 ^a	**	6.5	2.0	**	6.3	4.8	*	3.5	5.2	*
CS2	6.7	6.5 ^a	n.s.	5.3	3.0	*	6.7	3.5	**	3.2	3.5	n.s.
CS3	6.0	5.5 ^{ab}	n.s.	5.0	3.3	*	6.8	4.5	*	3.8	3.7	n.s.
Sign.	n.s.	**		n.s.	n.s.		n.s.	n.s.		n.s.	n.s.	

Small letters within a column show significant differences as assessed by Tukey's *post-hoc* test. Abbreviations: **, significance at $p < 0.01$; *, significance at $p < 0.05$; n.s., not significant. CTR: gummy candies without CSE; CS1: gummy candies with 1% CSE; CS2: gummy candies with 2% CSE; CS3: gummy candies with 4% CSE

Table 11 Taste of gummy candies at the beginning (1 day) and the end (120 days) storage period

Sample/ Days	Sweet			Fruity			Citrus			Astringent			Aftertaste			
	1	120	Sign.	1	120	Sign.	Sign.	1	120	Sign.	1	120	Sign.	1	120	Sign.
	CTR	6.9	5.5 ^{ab}	n.s.	6.4	6.7 ^{ab}	n.s.	n.s.	4.7	4.2	n.s.	1.8	2.2	n.s.	2.4	5.8
CS1	6.3	5.3 ^b	n.s.	6.2	5.5 ^b	n.s.	n.s.	5.0	3.8	n.s.	3.0	3.5	n.s.	3.8	5.5	*
CS2	6.5	6.5 ^a	n.s.	6.0	7.0 ^a	n.s.	n.s.	5.5	4.7	n.s.	2.8	2.7	n.s.	3.7	6.0	*
CS3	5.3	5.5 ^{ab}	n.s.	6.0	5.7 ^{ab}	n.s.	n.s.	3.5	4.8	*	2.8	3.0	n.s.	4.5	6.0	*
Sign.	n.s.	*		n.s.	*			n.s.	n.s.		n.s.	n.s.		n.s.	n.s.	

Small letters within a column show significant differences as assessed by Tukey's *post hoc* test. Abbreviations: **, significance at $p < 0.01$; *, significance at $p < 0.05$; n.s., not significant. CTR: gummy candies without CSE; CS1: gummy candies with 1% CSE; CS2: gummy candies with 2% CSE; CS3: gummy candies with 4% CSE

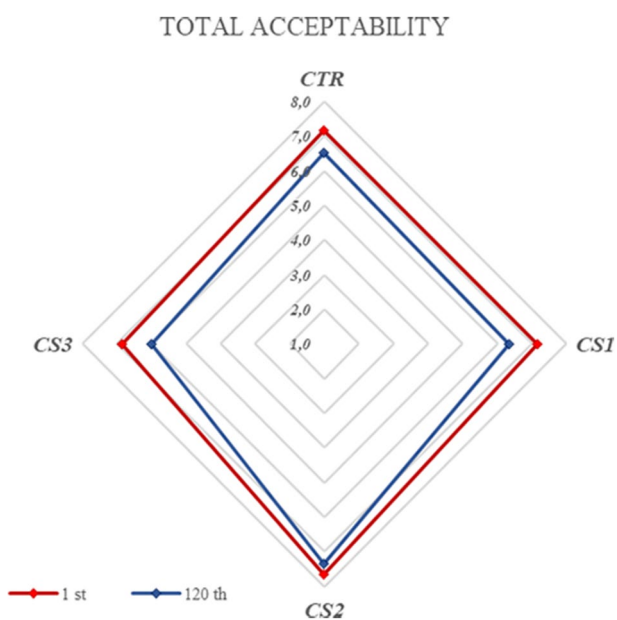


Fig. 3 Total acceptability of the gummy candies at the beginning (1 day) and the end (120days) storage period. (CTR: gummy candies without CSE; CS1: gummy candies with 1% CSE; CS2: gummy candies with 2% CSE; CS3: gummy candies with 4% CSE)

Conclusions

The addition of coffee silverskin has contributed positively to the chemical, physical, microbiological, and textural characteristics of candies. After 120 days of storage, the CS2 and CS3 candies showed the best results for antioxidant activity, and textural, and sensorial characteristics in comparison with CTR.

The results illustrated in this paper show that CSE could be a valid ingredient for the antioxidant enrichment of food products. The formulation of confectionery products with the aid of agri-food waste, as Coffee Silverskin, could have multiple advantages such as offering a vehicle of bioactive compounds for a large group of consumers, from children to adults, encouraging the transition of agri-food industries to a circular economy, enhance the production waste of the food sector and reduce environmental pollution resulting from the incorrect disposal of agri-food waste.

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Data availability Data will be made available upon request.

Declarations

Conflict of interest The authors declare no conflict of interest.

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