Powder Technology 440 (2024) 119777

Contents lists available at ScienceDirect

Powder Technology

journal homepage: www.journals.elsevier.com/powder-technology

Microencapsulated propolis powder: A promising ingredient of chewing gum

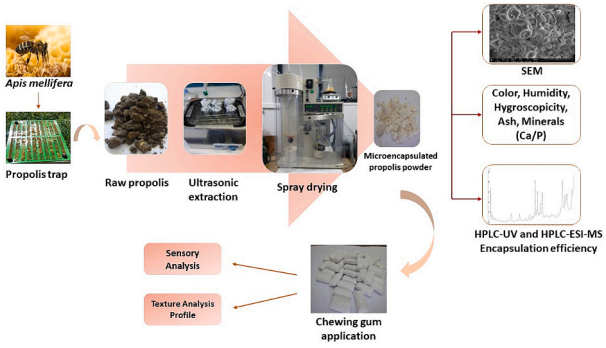
Wafa Gargouri [a], Mazen Elleuche [b], Miguel A. Fernández-Muiño [c], M. Teresa Sancho [c,*], Sandra M. Osés [c]

[a] Research Group of Agri-Food Processing Engineering, Laboratory of Applied Fluids Mechanics, Process Engineering and Environment "LR11ES57", National School of Engineers of Sfax, University of Sfax, Tunisia
[b] La Confiserie Triki le Moulin, Sfax, Tunisia
[c] Department of Biotechnology and Food Science, Universidad de Burgos (University of Burgos), Plaza Misael Bañuelos s/n 09001, Burgos, Spain

HIGHLIGHTS

- Spray drying encapsulation shows good retention of bioactive compounds.
- Phenolics of microencapsulated propolis powder are identified.
- Microencapsulated propolis powder has homogeneous surface without cracked walls.
- Propolis powder improves texture and sensory properties of chewing gum.
- Microencapsulated propolis powder can be used as functional food ingredient.

GRAPHICAL ABSTRACT



ARTICLE INFO

Keywords:
Propolis
Spray-drying
Chewing gum
Phenolics
Sensory quality

ABSTRACT

The incorporation of natural ingredients to food products as a source of bioactive compounds is currently an increasing trend for food companies. The purposes of this work were to study the physicochemical properties of microencapsulated propolis powder, and to research the potential beneficial effect of it on texture and sensory quality of sugar-free chewing gums. Propolis ethanolic extract was microencapsulated by spray-drying technology, using maltodextrin as carrier, protecting conceivable bioactivity. Morphology of propolis microparticles was evaluated using Scanning Electron Microscope (SEM) and showed homogeneous microparticles with different sizes and no cracks. Propolis powder showed low water activity, moisture and hygroscopicity, high amounts of Ca, P, and high values for antioxidant-related parameters. Propolis powder phenolics were analysed by HPLC-UV and HPLC-ESI-MS. Apigenin, gallic acid, CAPE, and galangin demonstrated the highest degree of encapsulation, providing antiradical activities. Propolis powder was incorporated in chewing gums at 5%, improving their texture and sensory properties.

* Corresponding author.
E-mail address: mtsancho@ubu.es (M.T. Sancho).

https://doi.org/10.1016/j.powtec.2024.119777
Received 6 March 2024; Received in revised form 12 April 2024; Accepted 13 April 2024
Available online 15 April 2024

1. Introduction

Propolis is a resinous substance, made by honeybees (*Apis mellifera*) by mixing the substances collected from trees, plant buds or exudates, and resins of different parts of plants with their saliva and beeswax. The chemical composition of propolis is given by the composition of the proximate plants [1]. It has been traditionally used in folk medicine to treat different diseases for its potential biological properties, among which, antioxidant, antimicrobial, anti-inflammatory, antihypertensive and anti-viral [2–4]. Propolis flavonoids and liposomes recently proved efficacy against SARS-COV-2 [5,6]. Consequently, there is a growing interest regarding propolis uses in the food industry to fulfil the current interest of consumers for natural and healthy foods [7].

Nowadays, there are different commercial products containing propolis such as candies, chocolates, tinctures and skin creams. However, the incorporation of this hive product as a food ingredient is limited by its alcohol solubility, strong flavour and aroma. Propolis encapsulation by spray-drying was proposed as an excellent procedure to prevent strong flavour and to protect bioactive substances [8]. Different food sectors are interested in ingredients' microencapsulation, such as encapsulated fish and flaxseed oils that were added to meat and dairy products to fortify them with omega-3 [9,10]. Pasrija et al. [11] also added microencapsulated green tea polyphenols to improve bread quality bakery and Abbasi et al. [12] incorporated microencapsulated citric acid into chewing gums to improve their sensory properties.

Due to its anti-inflammatory and antimicrobial properties, propolis has been widely used in oral and dental preparations such as mouthrinses, toothpastes and chewing gums [13]. Several studies demonstrated the efficacy of this natural substance in oral health by reducing caries [14], dental demineralization [15], gingival inflammations and dental infectious diseases [16].

There is a huge market for chewing gum, since it plays an essential role in the confectionery industry. Chewing gum is the most suitable medium for encapsulated bioactive compounds, because neither extreme heat, nor high moisture conditions are used during its making process. Several scientists incorporated propolis in chewing gum systems. Bölük et al. [17] evaluated the optimum amount of propolis that could be added to chewing gums regarding textural and sensorial characteristics. In the same context, Gargouri et al. [15] assessed the ability of chewing gums enriched with propolis to strengthen dental structure, enhancing its remineralization. Chewing gum is masticated in the mouth, so that bioactive compounds present in it can be absorbed efficiently in human body during chewing [18].

Considering the facts mentioned above, the objectives of this research were to obtain an encapsulated alcohol-free propolis powder by spray drying; to characterize this powder; to assess antiradical activities and retention of bioactive compounds and to incorporate it in sugar-free chewing gums, researching the effect of encapsulated propolis on sensory and textural parameters of the prepared chewing gums. To the best of our knowledge, this is the first research that assesses the sensory acceptability of chewing gums made with microencapsulated propolis powder, which is of utmost importance for their commercialization.

2. Materials and methods

2.1. Analytical standards and reagents

Catechin and gallic acid were purchased from Panreac (Barcelona, Spain). Galangin and caffeic acid phenethyl ester (CAPE) were obtained from TargetMol (Boston, USA). Pinocembrin, apigenin, kaempferol and chlorogenic acid were from Cymit Quimica, S.L. (Barcelona, Spain). Caffeic acid, p-coumaric acid, ferulic acid, naringenin, quercetin, Folin-Ciocalteau reagent, DPPH (2,2-diphenyl-1-picrylhydrazyl) and ABTS (2,2′-azino-bis(3-ethylbenzothiazoline-6-sulfonic acid diammonium salt) were purchased from Sigma–Aldrich (Steinheim, Germany). The reagents ethanol (96%), methanol (95%), sodium carbonate, nitric acid and hydrochloric acid were purchased from Fluka Chemie GmbH, part of Sigma-Aldrich (St Louis, MO, USA). Maltodextrin (dextrose equivalent 17) was purchased from Roquettes Frères (Lestrem, France). A Milli-Q water purification system (Millipore, part of Merck, Bedford, MA, USA) was used for deonization of the water used for the mobile phase and aetonitrile was of HPLC grade (VWR International Eurolab, Avantor, Llinars del Vallés, Cataluña, Spain).

2.2. Propolis sample and extract preparation

A sample of raw propolis was collected by local beekeepers from Beja (North Tunisia), in the spring of 2021. The collected propolis is known as brown poplar propolis (from *Populus sp.*). The sample was collected with a plastic propolis trap and then stored at −20 °C in dark conditions until use. Propolis sample was grounded in a marble mortar at −30 °C and extracts were prepared according to the method previously proposed by Gargouri et al. [2]. Briefly, 2.0000 g powder were extracted in the dark with 30 ml of 80% ethanol in an ultrasonic bath (Selecta, Abrera, Barcelona) with heating frequency of 40 KHz for 20 min. Then, the sample extract was filtered through Whatman No. 4 filter paper and subsequently stored in the dark at −20 °C until powder preparation.

2.3. Encapsulation of propolis extract by spray drying

Propolis extract was evaporated on a rotary evaporator at 40 °C to a final concentration of 0.2 g/ml. Encapsulation was done following the indications of da Silva et al. [19], with minor modifications. Carrier agent solution was prepared by stirring 30 g maltodextrin in 100 ml water (24 h), followed by homogenization by Ultra-Turrax 25 (IKA, Staufen, Germany) at 15000 rpm (2 min). Then, 17 ml of the concentrated propolis extract (0.2 g/ml) was added and homogenized by Ultra-Turrax (4 min). This amount was established after previous trials with different amount of propolis (0.1, 0.15, 0.2 and 0.3 g/ml). The resulting solution was spray dried in a mini spray dryer B-290 (BÜCHI Labortechnik AG, Flawil, Switzerland) with an inlet temperature of 130 °C, an outlet temperature of 80 ± 4 °C, an aspiration rate of 93% a Nozzle diameter of 1.5 mm and a pump flow rate of 10%. After drying, an alcohol-free powder was collected, placed in closed bottles, and kept in dry and dark conditions at room temperature. The powder was called MTP, microencapsulated Tunisian propolis.

2.4. Physicochemical properties of propolis microcapsules

2.4.1. Scanning electron microscopy (SEM)

Morphological analysis of microparticles was done using a Scanning Electron Microscope (SEM) [20]. Microparticles were arranged on the surface of a double-sided tape and coated with a thin layer of gold under vacuum with a balzers sputter coating device (JFC-1100E, JEOL, Tokyo, Japan). Images were captured at 2000× with a JEOL scanning electron microscope (JEOL, JSM-5400, Japan).

2.4.2. Colour

Propolis powder colour was measured with a Colour Flex spectrocolorimeter (CR-300 Chroma, Minolta, Japan). The instrument was standardized with standard white plates. CieLab coordinates of L* (lightness), a* (redness) and b* (yellowness) were recorded. The parameter L* was related to the clarity, a* to the red/green colour component and b* to the blue/yellow colour component.

2.4.3. Water activity (a_w) and moisture

Water activity of samples was determined by using a Rotronic Hygropalm apparatus (Rotronic Ag, Switzerland), which was adjusted at room temperature, varying from 25 to 30 °C. Moisture content was obtained by drying the sample at 105 ± 1 °C until constant weight.

2.4.4. Hygroscopicity

Hygroscopicity was measured with Cai and Corke [21] method with slight changes. Aliquots of 2.00 g of the spray dried powder were placed into Petri dishes at 25 °C in a hermetic plastic container filled with NaCl saturated solution (75% RH). After 7 days, the plates were weighed and the hygroscopicity was calculated as g of water absorbed per 100 g of dry powder (g/100 g).

2.4.5. Ash content and mineral composition

Ash content was evaluated by incinerating the powder in a muffle furnace at 550 °C for 4 h until complete combustion of the organic material [22]. For the analysis of calcium content, the incinerated product was treated with nitric acid and then with hot hydrochloric acid. The residue was filtered, and the resulting solution constituted the base extract for analysis (NF V18–108. 1984). The analysis was carried out by using an Analytic Jena ZEE 700 type atomic absorption spectrophotometer (Analytik Jena AG, Jena, Germany). For the determination of phosphorus content, the ashes were subjected to hot acid attack. Then, the residue was filtered and treated with the Vanado-molybdic reagent and analysed by a colorimetric method.

2.4.6. Microparticles breakage for the release of microencapsulated phenolic compounds

The procedure to break the spray dried propolis powder was carried out according to the method of dos Reis et al. [23]. Propolis powder (0.50 g) was mixed with 20 ml acidified methanol. The extract was stirred during 2 min, sonicated (Selecta, Abrera, Barcelone, Spain) during 15 min, and centrifuged (Thermo-scientific Centrifuge IEC CL30R, UK) during 5 min at 4000 rpm. The supernatant was eventually recovered.

2.5. Encapsulation efficiency determined using HPLC-UV and HPLC-ESI-MS to analyse phenolic composition of spray dried propolis powder

Propolis encapsulation efficiency was determined identifying and quantifying the phenolic compounds contained in both the propolis extract and the supernatant after the powder microparticles breakage. HPLC-UV analysis was carried out with a high-performance liquid chromatograph Varian Pro Star 310 (Varian, Victoria, Australia) [2]. A Microsorb-MV 100–5C 18 (150 × 4.6 mm, particle size 5 μm) was used as chromatographic column. Mixtures of 0.1% formic acid in Milli-Q water (A) and 0.1% formic acid in acetonitrile (B) were used as mobile phase with a mobile phase rate of 1 ml/ min and the following gradient: 0–7 min, 0% B, 7–12 min, 2% B, 12–20 min, 8% B, 20–23 min, 10% B, 23–33 min, 20% B, 33–45 min, 23% B, 45–50 min, 30% B, 50–55 min, 32% B, and 55–60 min, 50% B. The volume of sample injected was 20 μl. Detection was carried out at 280 nm.

The identification of unknown compounds was performed by comparing their ESI-MS fragmentation spectra with data from the literature [24–27] and with data from the Phenol-Explorer online chemical database (http://phenol-explorer.eu). For this purpose, a 1260 Infinity HPLC (Agilent Technologies Inc., Santa Clara, CA, USA) was used, connected to a quadrupole-time-fighting system (6545-Q-TOF) using the same column, mobile phase and flow conditions described for the previous HPLC-UV analysis. The ionisation method employed was electrospray ionisation (ESI) in the negative and positive ion mode with Dual AJS-ESI source using nitrogen as collision and nebulisation gas and with the following conditions: Gas temperature 325 °C, drying gas 10 l/ min, nebuliser 45 psi, Vcap 3500 V, nozzle voltage 200 V and cover gas at 350 °C. MS-TOF with fragmenter at 100 V, skimmer 45 V and OCT 1 RF VPP 750 V was used, acquiring data between 100 and 1000 *m/z*.

To quantify the various phenolic compounds, calibration curves of the standards (gallic acid, caffeic acid, catechin, chlorogenic acid, p-coumaric acid, ferulic acid, naringenin, quercetin, apigenin, kaempferol, pinocembrin, galangin and CAPE) were used at eight concentration levels covering the usual concentration levels of these compounds in propolis (0.0005–0.5 mg/ml). The linearity of all compounds was satisfactory, with r2 values >0.9925. For peaks where no standard was available, the quantification of the compound was expressed as caffeic acid.

The efficiency of encapsulation was determined by liquid chromatography. Indeed, this efficiency is defined as the ratio between the concentration of each compound detected in the dried powder (mg/g of dry powder), and the amount of this compound initially found in the Tunisian propolis ethanolic extract, previously extracted by ultrasounds [2]. The encapsulation efficiency was calculated by the following Eq. [8].

$$\%encapsulation = \frac{Xi \times 100}{Yi}$$

where *Xi* is the quantity of each compound found in the spray dried propolis powder (mg/g of dry powder), and Yi is the quantity of that compound initially present in the propolis extract entering the spray dryer (mg/g of propolis).

2.6. Antioxidant-related parameters

Total phenolics, total flavonoids as well as antiradical activities against $ABTS^+$ and DPPH free radicals were analysed in the supernatant after the rupture of microparticles.

2.6.1. Total phenolic content

Total phenolics were determined by using Folin-Ciocalteu method with some modifications [28]. Spray-dried propolis powder (10.00 mg) was mixed with 3 ml distilled water, 0.25 ml Folin-Ciocalteu reactive (0.2 N) and 2 ml sodium carbonate solution (75 g/l). The mixture was incubated 2 h in the dark, at room temperature. Then, absorbance was read at 760 nm, using a UV/Vis spectrophotometer V-630 (Jasco, Tokyo, Japan). Results were expressed as mg gallic acid (GA)/g propolis, using a range of GA for the calibration curve between 2 and 160 μg/ml.

2.6.2. Total flavonoids' content

Total flavonoids were determined according to the method proposed by Meda et al. [29], using quercetin as standard (5–250 μg/ml) and expressing the results as mg of quercetin (Q)/g propolis sample.

2.6.3. Evaluation of antiradical activities

Antiradical activities were assessed measuring the capability of the supernatant components to react with both $ABTS^+$ and DPPH free radicals.

$ABTS^+$ assay. Trolox equivalent antioxidant capacity (TEAC) of the propolis powder was carried out by the ABTS (3-ethylbenzothiazoline-6-sulphonic acid) radical cation decolorization assay [30,31] with minor modifications. An aliquot of 10 μl of the sample was mixed with 1490 μl of $ABTS^+$. The absorbance was read at 734 nm after 6 min. Trolox was used as standard for calibration curve (0.625–3 mM) and results were expressed as μmol trolox (T)/g propolis.

DPPH assay. The DPPH radical scavenging activity was determined according to Chaillou and Nazareno [32], with modifications. A volume of 50 μl of sample was mixed with 1.95 ml of an ethanolic DPPH solution. After 30 min of reaction, the absorbance of the mixture was recorded at 517 nm in a UV/Vis spectrophotometer V-530 (Jasco, Tokyo, Japan). A control was prepared by mixing all reagents but for the 95% ethanol which was used instead of the extract. Results were expressed as mg GA/g propolis.

2.7. Chewing gum preparation

Two different formulations of sugar-free chewing gums were prepared based on two types of flavours: liquorice and honey. Preparations without spray dried propolis powder were also made, being used as

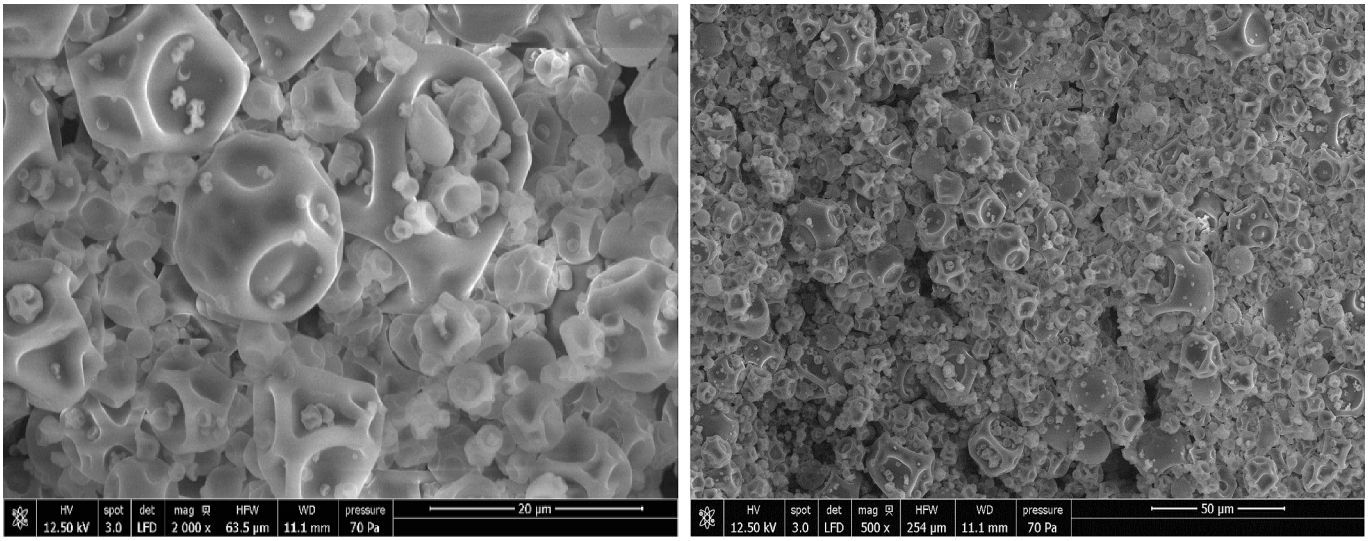


Fig. 1. Photomicrograph by SEM of the spray dried propolis microparticles by SEM at ×500 and ×2000 magnification.

controls. The chewing gums were prepared in the research and development laboratory of a Tunisian confectioner. The chewing gum ingredient formulation consisted of gum base that was melted in a chewing gum mixer (Tulpar Machine, Istanbul, Turkey) with heating at 40 °C. Then, the other ingredients, which were flavourings sweeteners (xylitol, sucralose and aspartame), colorants, humectants, emulsifying agents, were added together with the powder of the encapsulated propolis at a concentration of 5%.

2.8. Chewing-gum evaluation

2.8.1. Texture analysis profile (TPA)

Textural properties of the chewing gum samples (hardness, adhesiveness and fracturability) were determined using a TA1 texture analyser (LLOYD Instruments, England) with a 1000 (N) load cell and a detection range of 0.05 (N). The sample was compressed to 50% of its height (25 mm) at a speed of 25 mm/min with a cylindrical acrylic probe (diameter 0.3 mm). Data analysis was performed on a computer system coupled to the texture analyser using software supplied by Texture Technologies Corp.

2.8.2. Sensory analysis

The different groups of sugar-free chewing gums were evaluated by a well-trained panel of 10 assessors, aged 22–55 years (4 males and 6 females). Evaluators were located in individual cabins for sensory evaluation. The samples of chewing gums were similar in shape (rectangular), dimensions (10 × 20 × 4 mm), weight (1.4 g), and colour (white), so that subjects could not visually distinguish differences among them. The pieces of chewing gums were covered with aluminium foil and presented to the panelists in randomized order as coded samples. Subsequently, the organoleptic characteristics were quantified using five sensory attributes (flavour, aroma persistence, hardness, chewiness and overall quality). The first sensory test was for chewing gums with liquorice flavour and the second one was for those with honey flavour, with and without propolis powder. The assessors chewed each sample during 20 min. Then, they waited 15 min before chewing the following sample in order to minimize tiredness effect. Two sensory analyses were carried out using a 5-point scale, where 5 was “Excellent” and 1 “Not satisfactory”. Water and apples were served in order to clean the mouth between samples.

2.9. Statistical analysis

All analyses of physicochemical parameters, antiradical activities and texture were performed in triplicate and results of the study were expressed as average ± standard deviation. Statistical analysis was carried out with SPSS version 20 (SPSS Inc., Chicago, IL, USA). The data on sensorial analysis were assessed by *t*-test for two independent samples with significance level $p < 0.05$.

3. Results and discussion

3.1. Physico-chemical properties of propolis microcapsules

The micrographs of the microencapsulated Tunisian propolis (MTP) obtained by SEM, with 2000 × magnification, showed that the microcapsules exhibited a deformed spherical shape with bumpy surfaces, which was due to the loss of water from the particles during the spray drying process (Fig. 1). The forms of microcapsules did not affect encapsulation efficiency. The microcapsules of MTP were homogeneous presenting different sizes, which is a characteristic of microcapsules produced by spray drying. The observed structure was similar to that found in other studies, in which the properties of microcapsules of the propolis dry extract obtained by spray drying were analysed [8,33]. The absence of cracks and discontinuous walls at the surface of the microcapsules guaranteed a good barrier for the encapsulating agent and a better protection of the generated particles of the propolis powder than those obtained by other authors [8,19,23].

The visual appearance of the powder was pale yellow-coloured. This visual aspect was confirmed by the recording values of colour parameters. L* value was close to 100 (79.16 ± 0.44), which indicated a deviation towards the light shade. a* value was −0.11 ± 0.19. The positive value of b* (10.59 ± 0.76), showed a deviation towards the yellow, having the obtained powder a pale-yellow colour. Our colour parameters were in the same range to those obtained by Soleimanifard et al. [34] for Iranian spray dried propolis using caseinate-maltodextrin complexes as carrier agents.

The a_w of the MTP obtained was 0.40 ± 0.01. This value is characteristic of dehydrated food powders, being recommended for good storage stability [35]. Our a_w value was in the same range as that (0.44) of dos Reis et al. [23] for microencapsulated Brazilian propolis powder, being higher than the values (a_w between 0.25 and 0.33) found by da Silva et al. [19] in Brazilian propolis powder. These contrasting results can be explained by the fact that the a_w of propolis powder depends on

Table 1
HPLC peak assignation, retention time, area quantification and encapsulation efficiency of microencapsulated Tunisian propolis compounds.

Peak N°	Propolis compound	Retention time	Encapsulation efficiency (%)
1	Adipic acid†	1.8	47
2	Gallic acid	3.2	68
3	Caffeic acid	17.6	41
4	(+)- Catechin	18.3	0
5	Chrologenic acid	20.9	0
6	p-Coumaric acid	23.1	27
7	Ferulic acid	27.5	36
8	Isorhamnetin-3-O-rutinoside†	29.6	8
9	p-Coumaroyl malic acid†	33.4	17
10	Rutin†	34.3	14
11	Luteolin†	35	13
12	Pinobanksin†	37.15	0
13	Rosmarinic acid	38.4	34
14,15	Naringenin + Quercetin*	40.5	20
16	Isorhamnetin†	41.5	0
17	Apigenin	45.8	80
18	Kaempferol	46.3	54
19	Pinocembrin	51.3	28
20	Genistein†	52.2	45
21	Chrysin†	54.1	64
22,23	CAPE + Galangin*	55.5	65
24	4-Cinnamoyloxy caffeic acid†	58.8	51

* These compounds elute at the same RT, so they were quantified together.
† These compounds were quantified as mg of caffeic acid/g propolis.

several factors such as the nature of the encapsulating agent and the drying operating conditions.

The moisture percentage of the MTP (3.36 ± 0.14%) was lower than the values obtained by Andrade et al. [24] for Brazilian encapsulated propolis obtained with Arabic gum and maltodextrin as carrier agents, being similar to the results obtained by Kunrath et al. [36] for Italian propolis powder. Propolis spray dried powders with high moisture contents were less stable and can make their maintenance difficult during storage [37].

With regard to powder hygroscopicity, our powder was characterized by a low hygroscopicity 8.36% ± 0.11) in comparison with the range found for Argentinian (8.4–9.5%) and Brazilian (13.8%–29.3%) propolis powders, when using Arabic gum and corn products as carriers [8,19]. Our powder was easily handled and stored, exhibiting good stability, when it was stored in zipped plastic bags at room temperature during 6 months. Lower hygroscopicity could be attributed to the use of maltodextrin, since it was found that this carrier decreased the powder hygroscopicity and improved the powder quality when it was used in spray drying process [38,39]. In addition, the low hygroscopicity of the final powder could be also attributed to the composition of the initial propolis extract, characterized by negligible amounts of carbohydrate, thus providing a low hygroscopic powder [40].

Ash content of the MTP (10.65 ± 0.49%) was higher than the percentage found by Kunrath et al. [36] for Italian powder (3.12%). Percentage of ash is related to the mineral content of the dry propolis extract as well as the presence of impurities, due to the production process. In our propolis powder, high amounts of both calcium (1.39 ± 0.23 mg/g) and phosphorus (0.84 ± 0.03 mg/g) were found, so that the obtained propolis powder might be considered as a promising supplement of calcium and phosphorus. These two minerals are crucial for oral health. Different studies showed that the bioavailability of calcium and phosphorus is of utmost importance to produce a high level of remineralization [41,42].

3.2. Encapsulation efficiency of phenolic compounds

The efficiency of propolis compounds microencapsulation was assessed by analyzing phenolics in both MTP and the ethanolic propolis extract. Phenolics were quantified by HPLC-UV and identified by HPLC-ESI-MS (Table 1). Encapsulation efficiency (Table 1), varied depending on the phenolic compound. High encapsulation percentages were found for some compounds such as apigenin (80%), gallic acid (68%), CAPE+galangin (65%) and chrysin (64%). While for other compounds such as isorhamnetin-3-O-rutinoside (8%), luteolin (13%), rutin (14%) and p-coumaroyl malic acid (17%), low values were recorded. For the rest of compounds, the encapsulation efficiency varied from 20% to 54%, but non-quantifiable or non-detectable values were recorded for catechin (that was also absent in the extract), chlorogenic acid, pinobanksin and isorhamnetin, which were almost absent in the powder.

Busch et al. [8] studied the encapsulation of propolis from Argentina, using maltodextrin with or without Arabic gum as encapsulating agents. These researchers observed that the addition of gums to maltodextrin improved the encapsulation yield of certain phenolic compounds, but this yield did not exceed the percentage of 50%. However, in the present study, encapsulation using maltodextrin resulted in high encapsulation yields for several phenolic compounds, exceeding 50% for some of them. The high encapsulation efficiency could be attributed to the interaction between the bioactive compounds and the carrier, as well as to the operating conditions of the spray dryer, in particular regarding the temperature of the air drying. Indeed, the high temperature (around 120 °C) of the air flow demonstrated to have a positive impact on the encapsulation of bioactive compounds. The higher the drying temperature is, the more phenolic compounds are exposed, and therefore, the higher the evaporation rate is, generating a powder composed of many particles with larger contact surfaces [43,44]. It is very interesting to point out that the spray drying method generated an alcohol-free, high-quality propolis powder which preserved a large amount of the phenolic compounds initially present in the propolis ethanolic extract.

In the microencapsulated powder of propolis, phenolic compounds were researched. Considering the initial composition of the original extract and the results, the powder demonstrated to be very rich in CAPE, galangin and chrysin. Some antibacterial and antifungal properties were described for polyphenols. In a previous study with different propolis [2], we observed that the higher the amount of phenolics in propolis was, the higher the antibacterial activity was. Propolis also provide anti-inflammatory properties, which can be beneficial to periodontal health. Periodontal disease affects many adults globally and can result in painful, swollen, bleeding gums and in severe cases, tooth loss [45]. In recent studies, it has been proved that propolis, due to its polyphenol content principally CAPE and galangin, is able to inhibit cell viability within biofilms, decrease the total biomass of biofilms and disrupt biofilm structure of *Streptococcus mutans*, slowing bacterial growth and aiding prevention of dental caries [46,47].

3.3. Antioxidant-related parameters

Antioxidant capacity, as well as other biological activities, are mainly related to the phenolics' content and antiradical activities of propolis [3,30,48,49].

Total phenolics (29.82 ± 0.36 mg GA/g) and total flavonoids (1.32 ± 0.08 mg Q/g) of the MTP were in the same range as those found by Andrade et al. [24] for encapsulated Brazilian propolis when using maltodextrin as an encapsulating material (23.36–48.38 mg GA/g, for total phenolics and 1.34–2.76 mg Q/g, for total flavonoids). However, total phenolics were considerably lower than those found by Pratami et al. [50], for Indonesian propolis when using both maltodextrin and Arabic gum at different ratio as carrier agents (with results higher than 85.5 mg GA/g). This variation among propolis from several regions is related to the flora, geographical area, climate, season, as well as the type of extraction and solvents used [51].

Antiradical activities were determined by two different and general procedures against two different non-physiological free radicals ($ABTS^{+}$ and DPPH). The results of both rapid and simple analyses can help

Table 2
Textural properties of sugar-free chewing gum with and without propolis powder.*

	HC	HC + MTP	LC	LC + MTP
Fracturability (N)	14.03 ± 0.23[a]	8.93 ± 0.35[b]	12.70 ± 1.09[a]	6.34 ± 1.14[b]
Adhesiveness (N/mm)	−0.31 ± 0.02[a]	−0.98 ± 0.05[b]	−0.29 ± 0.01[a]	−0.98 ± 0.13[b]
Hardness (N)	14.9 ± 0.98[a]	10.25 ± 0.35[b]	15.15 ± 1.74[a]	11.74 ± 1.76[b]

* HC: Honey Chewing Gum; LC: Liquorice Chewing Gum; MTP: Microencapsulated Tunisian Propolis. Different lowercase letter in the same row, for the same chewing gum type indicates significant difference ($p < 0.05$) by the *t-test*.

characterize the propolis powder, notwithstanding the drawbacks of these procedures to evaluate biological antioxidant activities [52]. For $ABTS^{+}$ assay, the propolis spray-dried powder showed a TEAC of 8160.56 ± 10 μmol T/g. Our TEAC was similar than TEAC found by Andrade et al. [24], and higher than TEAC described by Reis et al. [23], being also higher than the results reported by Gargouri et al. [2] for propolis ethanolic extracts. DPPH method showed an antiradical activity value of 37.63 ± 1.14 mg GA/g, lower than the values obtained by Busch et al. [8] in propolis powders (ranging from 80 to 86 mg GA/g). In general, our propolis powder exhibited significant antiradical activities. Spray drying technology is known for its ability to protect antioxidant properties. Furthermore, the use of maltodextrin as an encapsulating agent demonstrated an important retention efficiency of bioactive compounds present in propolis ethanolic extract [8,53].

3.4. Chewing gum evaluation

3.4.1. Texture profile analysis (TPA)

Table 2 shows the effect of the addition of MTP on texture parameters of the prepared chewing gum.

Fracturability was defined as "the force at the first significant break in the curve", being adhesiveness "the negative force area of the first byte representing the work necessary to pull the compressing plunger away from the sample" [54]. Our values of fracturability decreased after the addition of MTP ($p < 0.05$). The decrease of fracturability is highly desirable in chewing gums. Indeed, high values of fracturability lead to a brittle product, whose structure could be destroyed during transport or storage. Conversely, our adhesiveness values increased after the addition of MTP.

Hardness was also lower in chewing gum containing MTP. According to Meullenet et al. [55], hardness corresponded to the "maximum force applied to a sample during the first compression cycle". Hardness was found to be the most important parameter affecting chewing gum's sensorial general acceptance [56]. The decrease of hardness of the gum when adding propolis microcapsules was also showed by Santos et al. [57] in chewing gum after adding microcapsules of xylitol and menthol. The behaviour of gum hardness towards the addition of microcapsules could be due to the large volume occupied by microcapsules with low density. Microcapsules require less energy in the chewing process. Therefore, the presence of propolis microcapsules in chewing gums is advantageous, because it improves their texture, diminishing fracturability and hardness.

3.4.2. Sensory evaluation

In our chewing gums microencapsulated propolis powder was incorporated at 5%, because at this percentage propolis had already proved to be highly accepted by consumers when it was added to fresh fish burgers [58]. In other studies [57,59], encapsulation systems showed to be effective to delay cooling flavours release with a longer pleasant perception.

Flavour, aroma persistence, hardness and chewiness were sensory researched. As the propolis taste is very bitter, liquorice and honey flavours were added in the formulation of chewing gums in order to improve their acceptability, because both liquorice and honey flavours could be compatible with propolis flavours. As shown in Fig. 2, for both honey and liquorice flavoured chewing gums, the sensory panel indicated a positive perception of hardness, chewiness, aroma persistence and overall quality of propolis added chewing gums. With regard to flavour, MTP improved the flavour of honey-flavoured chewing gums, but not the flavour of liquorice flavoured chewing gums ($p < 0.05$), so that liquorice flavour is not likely compatible with propolis flavour.

The addition of propolis in chewing gum formulation led to an improvement in both hardness and chewiness. Chewiness is one of the most important parameters for determining chewing gum sensory quality and is defined as the "total effort required to chew" [55]. Results of hardness and chewiness agreed with texture analysis, since the addition of propolis to chewing gum resulted in a decrease of the product hardness, making chewing gum softer and more appealing.

The panelists concluded that honey flavoured chewing gums made with microencapsulated propolis contained no sensory flaw that could jeopardize their commercialization.

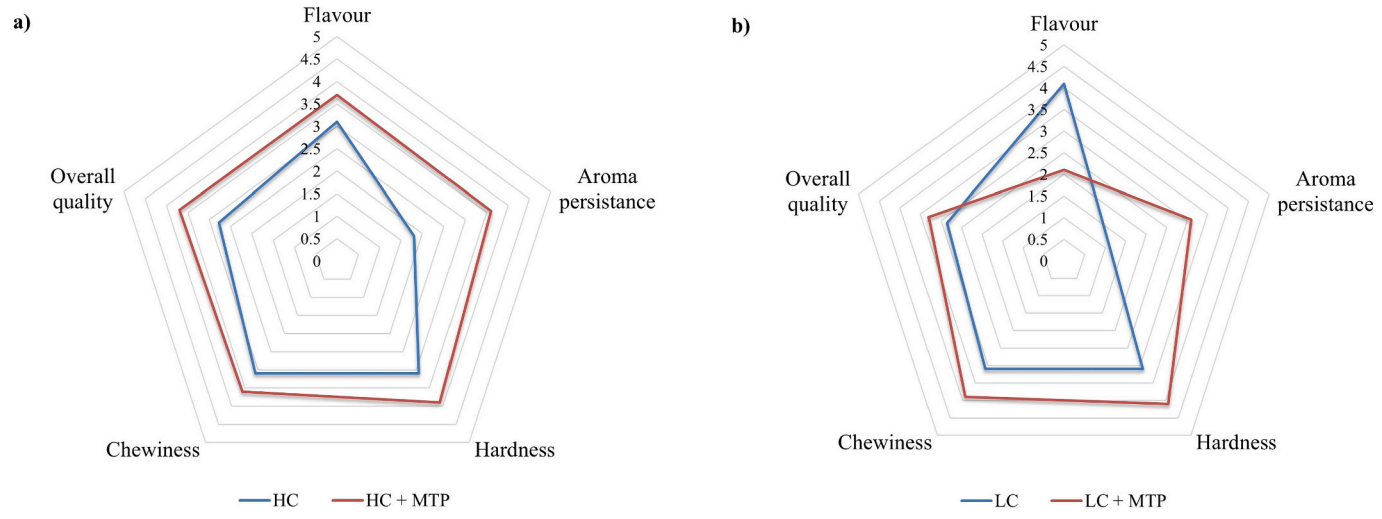


Fig. 2. Sensory evaluation of sugar-free honey (a) and liquorice (b) chewing gum with or without propolis powder (5 was Excellent and 1 was not satisfactory). HC: Honey Chewing Gum; LC: Liquorice Chewing Gum; MTP: Microencapsulated Tunisian Propolis.

4. Conclusion

The spray drying encapsulation of propolis powder proved to be efficient for providing a food ingredient with potentially beneficial characteristics. Microparticles were homogeneous. Propolis powder exhibited low water activity, moisture and hygroscopicity, high amounts of Ca and P and high results for antioxidant-related parameters. Propolis powder showed an interesting retention of the phenolic compounds of propolis ethanolic extract.

After adding 5% microencapsulated propolis powder to chewing gums, texture and sensory properties were improved in honey flavoured chewing gums in comparison with chewing gums made without propolis.

These findings suggest that the microencapsulated propolis as an ingredient of chewing gums could provide health benefits to oral cavity through the time of chewing, since propolis proved to be rich in bioactive compounds, also having a good antioxidant potential. Therefore, microencapsulated propolis might offer new applications as a food ingredient, because of its promising texture and sensorial properties. However, it is important to research the flavour compatibility of propolis to that of other foods, in order to choose the most suitable matrix for its incorporation.

CRediT authorship contribution statement

Wafa Gargouri: Writing – original draft, Validation, Investigation, Formal analysis, Data curation. **Mazen Elleuche:** Validation, Investigation, Formal analysis. **Miguel A. Fernández-Muiño:** Writing – original draft, Visualization, Supervision, Methodology, Investigation. **M. Teresa Sancho:** Writing – review & editing, Visualization, Supervision, Methodology, Investigation. **Sandra M. Osés:** Writing – original draft, Supervision, Resources, Methodology, Investigation, Data curation, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

Acknowledgments

Authors thank the valuable guidelines and supervision of Prof. Nabil Kechaou, who passed away before the writing of this research. This work was carried out under the MOBIDOC scheme, funded by The Ministry of Higher Education and Scientific Research through the PromESsE Project and managed by the ANPR. The authors are grateful to the Food and Technology Lab in the University of Burgos (Spain), for providing their premises and facilities.

References

[1] D.S. Dezmirean, C. Paşca, A.R. Moise, O. Bobiş, Plant sources responsible for the chemical composition and main bioactive properties of poplar-type propolis, Plants (Basel) 10 (2020) 22, https://doi.org/10.3390/plants10010022.
[2] W. Gargouri, S.M. Osés, M.A. Fernández-Muiño, M.T. Sancho, N. Kechaou, Evaluation of bioactive compounds and biological activities of Tunisian propolis, LWT Food Sci. Technol. 111 (2019) 328–336, https://doi.org/10.1016/j.lwt.2019.05.044.
[3] S.M. Osés, A. Pascual-Maté, M.A. Fernández-Muiño, T.M. López-Díaz, M.T. Sancho, Bioactive properties of honey with propolis, Food Chem. 196 (2016) 1215–1223, https://doi.org/10.1016/j.foodchem.2015.10.050.
[4] K. Pobiega, K. Kraśniewska, M. Gniewosz, Application of propolis in antimicrobial and antioxidative protection of food quality – A review, Trends Food Sci. Technol. (2019), https://doi.org/10.1016/J.TIFS.2018.11.007.
[5] A.M. Ali, H. Kunugi, Propolis, bee honey, and their components protect against coronavirus disease 2019 (COVID-19): a review of in silico, in vitro, and clinical studies, Molecules 26 (2021) 1232, https://doi.org/10.3390/molecules26051232.
[6] H. Refaat, F.M. Mady, H.A. Sarhan, H.S. Rateb, E. Alaaeldin, Optimization and evaluation of propolis liposomes as a promising therapeutic approach for COVID-19, Int. J. Pharm. 592 (2021) 120028, https://doi.org/10.1016/j.ijpharm.2020.120028.
[7] N. Shavisi, A. Khanjari, A.A. Basti, A. Misaghi, Y. Shahbazi, Effect of PLA films containing propolis ethanolic extract, cellulose nanoparticle and *Ziziphora clinopodioides* essential oil on chemical, microbial and sensory properties of minced beef, Meat Sci. 124 (2017) 95–104, https://doi.org/10.1016/j.meatsci.2016.10.015.
[8] V. Busch, A. Pereyra-Gonzalez, N. Šegatin, P. Santagapita, N. Ulrih, P. Buera, Propolis encapsulation by spray drying: characterization and stability, LWT Food Sci. Technol. 75 (2017), https://doi.org/10.1016/j.lwt.2016.08.055.
[9] A. Goyal, V. Sharma, M.K. Sihag, A.K. Singh, S. Arora, L. Sabikhi, Oxidative stability of alpha-linolenic acid (ω-3) in flaxseed oil microcapsules fortified market milk, Int. J. Pharm. 70 (2017) 188–196, https://doi.org/10.1111/1471-0307.12326.
[10] J.C. Solomando, T. Antequera, T. Perez-Palacios, Evaluating the use of fish oil microcapsules as omega-3 vehicle in cooked and dry-cured sausages as affected by their processing, storage and cooking, Meat Sci. 162 (2020) 108031, https://doi.org/10.1016/j.meatsci.2019.108031.
[11] D. Pasrija, P.N. Ezhilarasi, D. Indrani, C. Anandharamakrishnan, Microencapsulation of green tea polyphenols and its effect on incorporated bread quality, LWT Food Sci. Technol. 64 (2015) 289–296, https://doi.org/10.1016/j.lwt.2015.05.054.
[12] S. Abbasi, S. Rahimi, M. Azizi, Influence of microwave-microencapsulated citric acid on some sensory properties of chewing gum, J. Microencapsul. 26 (2009) 90–96, https://doi.org/10.1080/02652040802173677.
[13] S.G. Tulsani, N. Chikkanarasaiah, S.B. Siddaiah, N.H. Krishnamurthy, The effect of Propolis and xylitol chewing gums on salivary *Streptococcus mutans* count: a clinical trial, Indian J. Dent. Res. 25 (2014) 737, https://doi.org/10.4103/0970-9290.152182.
[14] M.F. Hayacibara, H. Koo, P.L. Rosalen, S. Duarte, E.M. Franco, W.H. Bowen, M. Ikegaki, J.A. Cury, In vitro and in vivo effects of isolated fractions of Brazilian propolis on caries development, J. Ethnopharmacol. 101 (2005) 110–115, https://doi.org/10.1016/j.jep.2005.04.001.
[15] W. Gargouri, R. Kammoun, M. Elleuche, M. Tlili, N. Kechaou, S. Ghoul-Mazgar, Effect of xylitol chewing gum enriched with propolis on dentin remineralization in vitro, Arch. Oral Biol. 112 (2020) 104684, https://doi.org/10.1016/j.archoralbio.2020.104684.
[16] B. Kouidhi, T. Zmantar, A. Bakhrouf, Anti-cariogenic and anti-biofilms activity of Tunisian propolis extract and its potential protective effect against cancer cells proliferation, Anaerobe 16 (2010) 566–571, https://doi.org/10.1016/j.anaerobe.2010.09.005.
[17] E. Bölük, D.S. Atik, S. Kolaylı, A.Ş. Demirci, I. Palabiyik, Delivery of phenolics and caffeic acid phenethyl ester by propolis resin: chewing gum system, Food Biosci. 41 (2021) 101090, https://doi.org/10.1016/j.fbio.2021.101090.
[18] N. Konar, I. Palabiyik, O.S. Toker, O. Sagdic, Chewing gum: production, quality parameters and opportunities for delivering bioactive compounds, Trends Food Sci. Technol. 55 (2016) 29–38, https://doi.org/10.1016/j.tifs.2016.07.003.
[19] F.C. da Silva, C.R. da Fonseca, S.M. de Alencar, M. Thomazini, J.C.C. de Balieiro, P. Pittia, C.S. Favaro-Trindade, Assessment of production efficiency, physicochemical properties and storage stability of spray-dried propolis, a natural food additive, using gum Arabic and OSA starch-based carrier systems, Food Bioprod. Process. 91 (2013) 28–36, https://doi.org/10.1016/j.fbp.2012.08.006.
[20] B. Shu, W. Yu, Y. Zhao, X. Liu, Study on microencapsulation of lycopene by spray-drying, J. Food Eng. 76 (2006) 664–669, https://doi.org/10.1016/j.jfoodeng.2005.05.062.
[21] Y.Z. Cai, H. Corke, Production and properties of spray-dried Amaranthus Betacyanin pigments, J. Food Eng. 65 (2000) 1248–1252, https://doi.org/10.1111/j.1365-2621.2000.tb10273.x.
[22] AOAC, Official Methods of Analysis, 18th edn, Association of Official Analytical Chemists, Arlington, VA, USA, 2005.
[23] A.S. dos Reis, C. Diedrich, C. de Moura, D. Pereira, J.F. de Almeida, L.D. da Silva, M.S.V. Plata-Oviedo, R.A.W. Tavares, S.T. Carpes, Physico-chemical characteristics of microencapsulated propolis co-product extract and its effect on storage stability of burger meat during storage at −15 °C, LWT Food Sci. Technol. 76 (2017) 306–313, https://doi.org/10.1016/j.lwt.2016.05.033.
[24] J.K.S. Andrade, M. Denadai, G.R.S. Andrade, C. da Cunha Nascimento, P. F. Barbosa, M.S. Jesus, N. Narain, Development and characterization of microencapsules containing spray dried powder obtained from Brazilian brown, green and red propolis, Food Res. Int. 109 (2018) 278–287, https://doi.org/10.1016/j.foodres.2018.04.048.
[25] K.M. Kasiotis, P. Anastasiadou, A. Papadopoulos, K. Machera, Revisiting Greek Propolis: chromatographic analysis and antioxidant activity study, PLoS One 12 (2017) e0170077, https://doi.org/10.1371/journal.pone.0170077.
[26] N. Nina, C. Quispe, F. Jiménez-Aspee, C. Theoduloz, A. Giménez, G. Schmeda-Hirschmann, Chemical profiling and antioxidant activity of Bolivian propolis, J. Sci. Food Agric. 96 (2016) 2142–2153, https://doi.org/10.1002/jsfa.7330.
[27] F. Sobral, R.C. Calhelha, L. Barros, M. Dueñas, A. Tomás, C. Santos-Buelga, M. Vilas-Boas, I.C.F.R. Ferreira, Flavonoid composition and antitumor activity of bee bread collected in Northeast Portugal, Molecules 22 (2017), https://doi.org/10.3390/molecules22020248.

[28] V.L. Singleton, R. Orthofer, R.M. Lamuela-Raventós, in: B.-M. in Enzymology (Ed.), [14] Analysis of Total Phenols and Other Oxidation Substrates and Antioxidants by Means of Folin-Ciocalteu Reagent, Academic Press, 1999, pp. 152–178. http://www.sciencedirect.com/science/article/pii/S0076687999990171.
[29] A. Meda, C.E. Lamien, M. Romito, J. Millogo, O.G. Nacoulma, Determination of the total phenolic, flavonoid and proline contents in Burkina Fasan honey, as well as their radical scavenging activity, Food Chem. 91 (2005) 571–577, https://doi.org/10.1016/j.foodchem.2004.10.006.
[30] S.M. Osés, P. Marcos, P. Azofra, A. de Pablo, M.Á. Fernández-Muíño, M.T. Sancho, Phenolic profile, antioxidant capacities and enzymatic inhibitory activities of Propolis from different geographical areas: needs for analytical harmonization, Antioxidants (Basel) 9 (2020) 75, https://doi.org/10.3390/antiox9010075.
[31] M.T. Sancho, A. Pascual-Maté, E.G. Rodríguez-Morales, S.M. Osés, I. Escriche, Á. Periche, M.A. Fernández-Muiño, Critical assessment of antioxidant-related parameters of honey, Int. J. Food Sci. Technol. 51 (2016) 30–36, https://doi.org/10.1111/ijfs.12988.
[32] L.L. Chaillou, M.A. Nazareno, Chemical variability in propolis from Santiago del Estero, Argentina, related to the arboreal environment as the sources of resins, J. Sci. Food Agric. 89 (2009) 978–983, https://doi.org/10.1002/jsfa.3542.
[33] J.F. de Almeida, S.T. Carpes, M.S.V.P. Oviedo, L.F. Serafini, D. Pereira, Antioxidant activity in vitro and microencapsulation by spray drying of hydroalcoholic extract of sage (*Salvia officinalis* L.), Braz. J. Food Res. 5 (2014) 41–49, https://doi.org/10.14685/rebrapa.v5i2.170.
[34] M. Soleimanifard, J. Feizy, F. Maestrelli, Nanoencapsulation of propolis extract by sodium caseinate-maltodextrin complexes, Food Bioprod. Process. 128 (2021) 177–185, https://doi.org/10.1016/j.fbp.2021.05.005.
[35] P.R. Bajaj, J. Tang, S.S. Sablani, Pea protein isolates: Novel Wall materials for microencapsulating flaxseed oil, J. Food Technol. 8 (2015) 2418–2428, https://doi.org/10.1007/s11947-015-1589-6.
[36] C. Kunrath, D. Savoldi, J. Mileski, C. Novello, A. Alfaro, J. Marchi, I. Tonial, Application and evaluation of propolis, the natural antioxidant in Italian-type salami, J. Food Technol. 20 (2017), https://doi.org/10.1590/1981-6723.3516.
[37] A.L. Siqueira, C.G. Dantas, M.Z. Gomes, F.F. Padilha, R.L.C. de Albuquerque Junior, J.C. Cardoso, Study of antibacterial action of hydroalcoholic extract of propolis red on *Enterococcus faecalis*, Rev. Odontol. UNESP 43 (2014) 359–366, https://doi.org/10.1590/1807-2577.1005.
[38] A. do Valle Calomeni, V.B. de Souza, F.L. Tulini, M. Thomazini, L.C. Ostroschi, S. M. de Alencar, A.P. Massarioli, J.C. de Carvalho Balieiro, R.A. de Carvalho, C. S. Favaro-Trindade, Characterization of antioxidant and antimicrobial properties of spray-dried extracts from peanut skins, Food Bioprod. Process. 105 (2017) 215–223, https://doi.org/10.1016/j.fbp.2017.08.001.
[39] G.R. Rodríguez-Hernández, R. González-García, A. Grajales-Lagunes, M.A. Ruiz-Cabrera, M. Abud-Archila, Spray-drying of Cactus pear juice (*Opuntia streptacantha*): effect on the physicochemical properties of powder and reconstituted product, Dry. Technol. 23 (2005) 955–973, https://doi.org/10.1080/DRT-200054251.
[40] K. Pant, M. Thakur, H.K. Chopra, V. Nanda, Encapsulated bee propolis powder: drying process optimization and physicochemical characterization, LWT 155 (2022) 112956, https://doi.org/10.1016/j.lwt.2021.112956.
[41] F.G. de Carvalho, V.L.M. Brasil, T.J. da Silva Filho, H.L. Carlo, R.L. dos Santos, B.A. S.G. de Lima, Protective effect of calcium nanophosphate and CPP-ACP agents on enamel erosion, Braz. Oral Res. 27 (2013) 463–470, https://doi.org/10.1590/S1806-83242013000600004.
[42] P. Shen, G.D. Walker, Y. Yuan, C. Reynolds, D.P. Stanton, J.R. Fernando, E. C. Reynolds, Importance of bioavailable calcium in fluoride dentifrices for enamel remineralization, J. Dent. 78 (2018) 59–64, https://doi.org/10.1016/j.jdent.2018.08.005.
[43] M. Paini, B. Aliakbarian, A.A. Casazza, A. Lagazzo, R. Botter, P. Perego, Microencapsulation of phenolic compounds from olive pomace using spray drying: A study of operative parameters, LWT Food Sci. Technol. 62 (2015) 177–186, https://doi.org/10.1016/j.lwt.2015.01.022.
[44] U. Baysan, A. Zungur Bastıoğlu, N.Ö. Coşkun, D. Konuk Takma, E. Ülkeryıldız Balçık, H. Sahin-Nadeem, M. Koç, The effect of coating material combination and encapsulation method on propolis powder properties, LWT Food Sci. Technol. 384 (2021) 332–341, https://doi.org/10.1016/j.powtec.2021.02.018.
[45] S. Petti, C. Scully, Polyphenols, oral health and disease: A review, J. Dent. 37 (2009) 413–423, https://doi.org/10.1016/j.jdent.2009.02.003.
[46] J. Yuan, W. Yuan, Y. Guo, Q. Wu, F. Wang, H. Xuan, Anti-biofilm activities of Chinese poplar Propolis essential oil against *Streptococcus mutans*, Nutrients 14 (2022) 3290, https://doi.org/10.3390/nu14163290.
[47] A. Kurek-Górecka, K. Walczyńska-Dragon, R. Felitti, S. Baron, P. Olczyk, Propolis and diet rich in polyphenols as Cariostatic agents reducing accumulation of dental plaque, Molecules 27 (2022) 271, https://doi.org/10.3390/molecules27010271.
[48] R. Cauich-Kumul, M.R. Segura Campos, Chapter 12 - bee Propolis: properties, chemical composition, applications, and potential health effects, in: M.R.S. Campos (Ed.), Bioactive Compounds, Woodhead Publishing, 2019, pp. 227–243, https://doi.org/10.1016/B978-0-12-814774-0.00012-8.
[49] S.M. Osés, L. Melgosa, A. Pascual-Maté, M.A. Fernández-Muiño, M.T. Sancho, Design of a food product composed of honey and propolis, J. Apic. Res. 54 (2015) 461–467, https://doi.org/10.1080/00218839.2016.1183934.
[50] D.K. Pratami, A. Mun'im, M. Yohda, H. Hermansyah, M. Gozan, Y.R.P. Putri, M. Sahlan, Total phenolic content and antioxidant activity of spray-dried microcapsules propolis from Tetragonula species, AIP Conf. Proc. 2085 (2019) 020040, https://doi.org/10.1063/1.5095018.
[51] V.D. Wagh, Propolis: a wonder bees product and its pharmacological potentials, Adv. Pharmacol. Pharm. Sci. 2013 (2013) e308249, https://doi.org/10.1155/2013/308249.
[52] K.M. Schaich, X. Tian, J. Xie, Hurdles and pitfalls in measuring antioxidant efficacy: A critical evaluation of ABTS, DPPH, and ORAC assays, J. Funct. Foods 14 (2015) 111–125, https://doi.org/10.1016/j.jff.2015.01.043.
[53] L. Šturm, I.G. Osojnik Črnivec, K. Istenič, A. Ota, P. Megušar, A. Slukan, M. Humar, S. Levic, V. Nedović, R. Kopinč, M. Deželak, A. Pereyra Gonzales, N. Poklar Ulrih, Encapsulation of non-dewaxed propolis by freeze-drying and spray-drying using gum Arabic, maltodextrin and inulin as coating materials, Food Bioprod. Process. 116 (2019) 196–211, https://doi.org/10.1016/j.fbp.2019.05.008.
[54] M.C. Bourne, S.H. Comstock, Effect of degree of compression on texture profile parameters, J. Texture Stud. 12 (1981) 201–216, https://doi.org/10.1111/j.1745-4603.1981.tb01232.x.
[55] J.-F. Meullenet, B.G. Lyon, J.A. Carpenter, C.E. Lyon, Relationship between sensory and instrumental texture profile attributes, J. Sens. Stud. 13 (1998) 77–93, https://doi.org/10.1111/j.1745-459X.1998.tb00076.x.
[56] I. Palabiyik, T. Güleri, R. Gunes, B. Öner, O.S. Toker, N. Konar, A fundamental optimization study on chewing gum textural and sensorial properties: the effect of ingredients, Food Struct. 26 (2020) 100155, https://doi.org/10.1016/j.foostr.2020.100155.
[57] M.G. Santos, D.A. Carpinteiro, M. Thomazini, G.A. Rocha-Selmi, A.G. da Cruz, C.E. C. Rodrigues, C.S. Favaro-Trindade, Coencapsulation of xylitol and menthol by double emulsion followed by complex coacervation and microcapsule application in chewing gum, Food Res. Int. 66 (2014) 454–462, https://doi.org/10.1016/j.foodres.2014.10.010.
[58] S. Spinelli, A. Conte, L. Lecce, A.L. Incoronato, M.A. Del Nobile, Microencapsulated Propolis to enhance the antioxidant properties of fresh fish burgers, J. Food Process Eng. 38 (2015) 527–535, https://doi.org/10.1111/jfpe.12183.
[59] K. Sostmann, R. Potineni, G. Blancher, X. Zhang, M. Espinosa-Díaz, Aroma release from encapsulation systems in chewing gum, 2010. https://www.semanticscholar.org/paper/AROMA-RELEASE-FROM-ENCAPSULATION-SYSTEMS-IN-CHEWING-Sostmann-Potineni/128ac5cec63fa2db590d6a2007336d78f2a6b278 (accessed January 25, 2022).