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Microencapsulation of Phenolic Compounds Extracted from Okra (*Abelmoschus esculentus* L.) Leaves, Fruits and Seeds

Salma Guebebia [1], Adem Gharsallaoui [2], Emilie Dumas [2], Fatemeh Baghi [2,3], Lazhar Zourgui [4], Mehrez Romdhane [1], Géraldine Agusti [2] and Sami Ghnimi [2,3,*]

[1] Laboratory of Environment, Catalysis and Process Analysis (LEEEP), National School of Engineers of Gabes (ENIG), University of Gabes, Medenine Road, Gabes 6029, Tunisia; salmaguebebia1992@gmail.com (S.G.)
[2] LAGEPP, Université Claude Bernard Lyon 1, UMR 5007, F-69622 Villeurbanne, France
[3] ISARA, Higher Institute of Agriculture and Agri-Food Rhone-Alpes, 23 Rue Jean Baldassini, F-69007 Lyon, France
[4] Laboratory of Active Biomolecules Valorisation, Department of Biological Engineering, Research Unit of, Higher Institute of Applied Biology of Mednine (ISBAM), University of Gabes, Gabes 6029, Tunisia
* Correspondence: sami.ghnimi@univ-lyon1.fr or sghnimi@isara.fr; Tel.: +33-(0)4-27-85-86-70

Abstract: Several plants rich in phenolic compounds have many uses in the food and pharmaceutical fields. However, after extraction, these active biomolecules are susceptible to degradation. Microencapsulation is a possible solution to prevent this rapid degradation. In this study, phenolic compounds from the okra *Abelmoschus esculentus* L's leaves, fruits and seeds were extracted using microwave-assisted extraction and then microencapsulated via the spray drying technique using maltodextrin combined with pectin (in a ratio of 10:1) as an encapsulation material. The total phenolic content, DPPH scavenging and antimicrobial activities of okra extracts and encapsulated samples were evaluated to verify the encapsulation efficiency. Particle size distribution determination and scanning electron microscopy of the microcapsules were also carried-out. The ethanolic leaf extract showed higher significant levels of total phenolic compounds (162.46 ± 4.48 mg GAE/g DW), and anti-oxidant (75.65%) and antibacterial activities compared to those of other aqueous and ethanolic extracts from fruits and seeds. Furthermore, the spray-dried ethanolic leaf extract had the highest total phenolic content. However, the encapsulated ethanolic fruit extract had the highest percentage of DPPH scavenging activity (30.36% ± 1.49). In addition, antibacterial activity measurements showed that the addition of ethanolic and aqueous seed microcapsules provided a significant zone of inhibition against the bacterium *Brochotrix thermosphacta* (38 mm and 30 mm, respectively). Okra aqueous leaf microcapsules showed the smallest Sauter mean diameter values (7.98 ± 0.12 µm). These data are applicable for expanding the use of okra leaves, fruits and seeds as food additives and/or preservatives in the food industry.

Keywords: *Abelmoschus esculentus* L.; microencapsulation; phenolic compounds; antibacterial activity; microcapsules; spray-drying

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1. Introduction

Currently, there is an increased interest in the analysis of medicinal plants used as functional foods and herbal medicines. Scientific research has developed exponentially in order to evaluate their constituents in secondary metabolites thanks to their applications as pharmaceuticals and food additives [1,2]. However, phenolic compounds are the largest family of secondary metabolites. Therefore, medicinal plants are crucial sources of biological activities [3].

Okra (*Abelmoschus esculentus* L.), also known as lady's finger, is an annual herbaceous vegetable crop plant belonging to the Malvaceae family. It is widely cultivated in Africa,

Asia, southern Europe and the Americas [4]. Okra is basically used as an important vegetable thanks to its high vitamin and mineral content, dietary fiber, calcium content and low saturated fat content [5]. In addition, considering its photochemical compounds, okra has several bioactivities [6] such as anti-oxidant, immunomodulatory, anti-diabetic [7], anti-obesity [8], anti-cancer [3], anti-inflammatory, laxative, anti-hyperlipidemic, anti-fungal, analgesic, neuroprotective and anti-fatigue activities [9]. Different parts of *Abelmoschus esculentus* L. (leaves, buds, flowers, pods, stems and seeds) have many applications where the plant is considered a versatile crop [10]. Further studies reported that okra fruits have been used as a transitional medicines around the world due to their high content of polysaccharides and phenolic compounds, including flavonoids, which provide health-promoting benefits [11]. Similarly, okra leaves are also suitable for medicinal and industrial uses due to their polyphenolic compounds, and are often used for their biological activities [12]. Thus, okra leaves contain natural anti-oxidants. In addition, the whole okra plant has medicinal applications, but leaves are the richest part of the plant in oligomeric catechins and flavonol derivatives with anti-oxidant properties [13]. Globally, the major bioactive components of okra responsible for its several biological activities are polysaccharides and phenolic compounds [6]. Due to its excellent bioactivities and medicinal properties, recently, *Abelmoschus esculentus* L. has been the subject of various studies. It is ordinarily considered that *Abelmoschus esculentus* L. could be used as a natural anti-oxidant and antibacterial agent in foods and pharmaceutical domains [13,14]. To have optimal efficiency, biomolecules must be in an appropriate form. Globules and microparticles are considered the most suitable form [15]. However, the phenolic content of Tunisian okra leaf, fruit and seed has not been well known until now. Therefore, research is needed to analyze its phytochemical content, anti-oxidant and bactericidal proprieties, which may have healthcare benefits for the community.

Nevertheless, the phenolic compounds are influenced by external factors like light, temperature, pH, water and enzymatic activities [16,17]. Therefore, the stability and durability of phenolic compounds should be protected from chemical and physical damage prior to their industrial application. Microencapsulation is an approach in which an active component is encapsulated by a protective layer at the microscale [18]. It is one of the techniques commonly used in the food industry to protect biomolecules extracted from various natural sources. From this perspective, there are various encapsulation methods [19]. Among them, spray drying is one of the most frequently selected process due to its low cost and flexibility. In addition, the drying parameters and the coating material usually affect the retention capacity of the encapsulated compounds within the matrix. Maltodextrins are one of the main wall materials used as microencapsulation agents [20–22]. It is a nearly economical polysaccharide with a neutral taste and aroma, and an effective coating material for protection [23]. In addition, pectin is one of the most commonly used encapsulating agents in the spray drying process. Thanks to its low cost, pectin can be economically used as a wall material in the microencapsulation process [24]. Indeed, it has been reported in the literature that maltodextrin and pectin are used alone or in combination with other materials in the food and pharmaceutical processing of plant extracts to microencapsulate polyphenolic extracts [25].

In terms of the literature, there are numerous articles published on the extraction of okra, but to our knowledge, no study is available on the encapsulation of okra extracts for food applications. Moreover, no study has been published on the extraction and encapsulation of Tunisian okra.

Therefore, the objective of the present study was to extract Tunisian okra polyphenols, determine their anti-oxidant and antibacterial activities, and then encapsulate them by spray drying them using maltodextrin and pectin as coating materials. This study highlighted the effect of the solvent used and the effect of the plant organ studied on phenolic contents and their bioactivities. The physicochemical properties of the obtained microcapsules were also investigated in terms of encapsulation efficiency, total phenolic content, particle size, microstructure, anti-oxidant and antibacterial activities.

2. Materials and Methods

2.1. Materials

The green okra *Abelmoschus esculentus* L leaves, fruits and seeds used in this study were collected in July–August 2020 from a local farm (Gabès, Tunisia), while leaves and seeds of matured okra were randomly collected from the same farm in October 2020 and taken to the laboratory. Okra fruits were then washed thoroughly with distilled water to remove foreign matter and cut into 5 mm pieces. They were dried at 40 °C for 6 days until their mass stabilized. Okra leaves and seeds were then washed with distilled water and dried. After that, samples were ground and sieved into a fine powder with a 425 µm sieve (Endecotts, London, UK). The powders were then stored in the dark at 4 °C prior to analysis. Powdered pectin and maltodextrin (DE28) were obtained from Roquette-frères SA (Lestrem, France). The reagent used in the experiments, which were acetic acid, ethanol, methanol, gallic acid, DPPH (2,2-diphenyl1picrylhydrazyl), Folin-Ciocalteau phenol reagent and glycerol were purchased from Sigma-Aldrich (St Quentin Fallavier, France).

2.2. Moisture Content

The standard official method of analysis of the AOAC was used to determinate the moisture content of the samples [26].

2.3. Extraction of Phenolic Compounds

The extraction was performed following the procedure of ElïK et al. [27] with some modifications. Aqueous and ethanolic extracts were prepared using microwave-assisted extraction. Briefly, samples of dried okra powder (1 g) were mixed with 25 mL of solvent (distilled water or 70% ethanol) for 2 min at 100 W (at 37 °C for aqueous extracts and 42 °C for ethanolic extracts). After that, all the extracts obtained were cooled to room temperature and centrifuged at 8000× *g* for 10 min. The supernatants were filtered through a 0.2 µm solvent filter and stored at 4 °C until use. The extraction yield (Y), defined as the ratio between the mass obtained from the dry extract and the mass of the treated plant material, was calculated using the following equation:

$$Yield(\%) = \frac{m_f}{m_i} \times 100 \quad (1)$$

where m_f is the mass of the extract after the evaporation of the solvent and m_i is the mass of the plant material used for extraction.

2.4. Phenolic Powder Preparation

The filtered ethanolic extract was concentrated at 45 °C using a rotary vacuum evaporator (Rotavapor R-300, BUCHI, Flawil, Switzerland) until its volume nearly decreased to almost 1/3 of the initial value. The samples were then dried using a freeze dryer (L-200 pro, BUCHI, Switzerland) and the resulting freeze-dried samples of okra fruits, leaves and seeds were ground into fine powder form and stored in the freezer until analysis.

Okra extracts were concentrated and then freeze-dried to remove ethanol (traces of alcohol) to highlight their antibacterial effects.

2.5. Encapsulation

The encapsulation assays were conducted as follows: for 100 g of solution, 1 g of dried phenolic okra powder was dissolved in distilled water at room temperature until a homogeneous solution was obtained. It was then combined with MD solution (10% *w*/*w*) alone under constant stirring, and finally pectin solution (1% *w*/*w*) was added. The combination was homogenized using a magnetic stirrer at 300 rpm for 1 h at 25 °C. The samples were then subjected to a spray drying process using a mini spray dryer (Büchi B-290, Flawil, Switzerland) under the following experimental conditions: drying air inlet temperature: 150 °C; outlet temperature: 75 °C; and feed rate: 500 mL/h. The diameter of the spray nozzle was 0.5 mm. The same settings were used for all the extracts (leaves,

seeds and fruits). The resulting powders were stored away from light at −20 °C until the analysis of the encapsulated powders was performed.

2.6. Analysis of Phenolic Powder and Encapsulated Phenolic Powder

Spray-dried powders were rehydrated until the same soluble solid content as measured before drying was reached prior to their analysis.

2.6.1. Total Phenolic Content

The total phenolic content (TPC) of the uncoated phenolic extract was estimated adopting the Folin–Ciocalteu method as described by Singleton et al. [28] with a simple adjustment. For the coated phenolic powder, approximately 120 mg of encapsulated phenolic powder was precisely weighed and dissolved in 1 mL of an ethanol:acetic acid:water (50:8:42) mixture [29]. The absorbance at 765 nm was measured using a UV-visible spectrophotometer (UV-3100PC, VWR, Radnor, PA, USA) and the total phenolic content of the extracts (TPC) and microcapsules (TPCC) was calculated using gallic acid calibration curves and expressed as gallic acid equivalents (mg GAE/g dry weight (DW)).

2.6.2. Total Anti-Oxidant Activity

The DPPH method, based on radical scavenging activity, was carried out for the extracts in accordance with the protocol described by Velázquez et al. [29]. Briefly, 0.75 mL of each okra extract was mixed with 1.5 mL of DPPH solution in 90% methanol (20 mg L^{-1}) and microcapsules were dissolved in 1 mL of an ethanol:acetic acid:water (50:8:42 v/v) mixture using Vortex for 1 min. Then, 0.75 mL of each sample (extract and microcapsules) was mixed with 1.5 mL of DPPH solution in methanol 90% (20 mg/L). After incubation for 15 min at room temperature, the scavenging activities of the extract, microcapsules and reference (Pure methanol) were evaluated by measuring the absorbance at 517 nm using a spectrophotometer (UV-3100PC, VWR, Radnor, USA). All tests were performed in triplicate. The anti-oxidant activity was expressed as a percentage of inhibition according to Equation (2):

$$I(\%) = \frac{A_0 - A_S}{A_0} \times 100 \quad (2)$$

where A_0 is the absorbance of the control and A_S is the absorbance of the sample.

2.6.3. Surface Phenolic Content (SPC) of Microcapsules

For the analysis of SPC, the Folin–Ciocalteau approach was used in the same way as it was for the TPCC content [30]. The exclusive difference was the dispersal of 120 mg of microcapsules in 1 mL of an ethanol:methanol mixture (50:50 v/v). SPC content was calculated using the gallic acid calibration curve and expressed in mg GAE/g DW.

2.6.4. Encapsulation Efficiency

Encapsulation efficiency (EE) is determined by the difference between the total phenolic content (TPCC) of microcapsules and the surface phenolic content (SPC) of microcapsules. The encapsulation efficiency of microcapsules was calculated in accordance with Equation (3) [30]:

$$EE(\%) = \frac{EPC}{TPC} \times 100 = \frac{TPCC - SPC}{TPCC} \times 100 \quad (3)$$

2.6.5. Particle Size Analysis

Particle size measurements were realized using a laser diffraction instrument (Malvern Mastersizer 3000, Malvern, Worcestershire, UK). Prior to the measurements, the microparticles were diluted with absolute ethanol in order to avoid scattering consequences and to prevent their dissolution. The microcapsules were stirred continuously throughout the

measurement to ensure the homogeneity of the samples. The volume particle diameter (D [4,3]) and the span values were calculated in accordance with Equations (4) and (5) [31]:

$$D[4,3] = \frac{\sum n_i d_i^4}{\sum n_i d_i^3} \quad (4)$$

$$\text{Span} = \frac{[d(v,90) - d(v,10)]}{d(v,50)} \quad (5)$$

where d_i is the diameter, n_i is number of particles in each size and $d(v, 10)$, $d(v, 50)$, and $d(v, 90)$ are the diameter values in the order of 10, 50, and 90% of the cumulative volume.

2.6.6. Microstructural Analysis

Scanning electron microscopy (FEI Quanta 250 microscope (Eindhoven, The Netherlands)) was used to perform (morphological) microstructural analysis at the "Centre Technologique des Microstructures" (CTμ) at the University of Claude Bernard Lyon 1 (Villeurbanne, France). The samples were coated under a vacuum via cathodic sputtering before performing microscopy analysis.

2.7. Antibacterial Activity

The antibacterial activity of uncoated plant extracts was evaluated against four reference bacteria: *Brochotrix thermosphacta* DSM20171, *Listeria Innocua* DSM20640 (two Gram-positive bacteria), *Escherichia coli* DSM613 and *Salmonella enterica* DSM11320 (two Gram-negative bacteria). The strains were stored at −20 °C in tryptone soy broth (TSB) (Biokar diagnostics, Beauvais, France) with glycerol (15 mL/100 mL). Then, 1mL of the stock culture was transferred to 9 mL of TSB and incubated for 8 h at 37 °C. One mL of this pre-culture was transferred in 9 mL of TSB and incubated at 37 °C for 16 h. The pre-cultured bacteria were then diluted in TSB to a final concentration of 10^6 CFU/mL. Briefly, 1 mL of the bacterial suspension was incorporated into melted tryptone soy agar (TSA) in an amount of 5 mL/100 mL and cooled in Petri dishes. A sterilized glass pipette (6 mm diameter, Fisher Scientific, Hampton, NH, USA) was used to inoculate a well in the TSA Petri dishes. Under aseptic conditions, the wells were impregnated with 100 μL of uncoated phenolic compounds (10 mg/mL) and placed on the culture plates after removing the solvent via evaporation. Physiological water was used as a negative control. The Petri dishes were then kept at 37 °C for 24 h. Antibacterial activity was assessed by measuring the zone of growth inhibition (mm) around the well. All experiments were performed in triplicate.

Similarly, the antibacterial activity of all microcapsules against four bacteria was carried out using the agar well diffusion method. The antibacterial effect of the microparticles was determined using the protocol previously used [32]. An amount of 12 mg of microcapsule powder was added to each well. The Petri dishes were then incubated at 4 °C for 2 h and then at 37 °C. Finally, the diameter of the inhibition zone was measured after 24 h and each experiment was repeated three times.

2.8. Statistical Analysis

All tests were performed at least in triplicate. The data were presented as means ± standard deviations for different samples. One-way analysis of variance (ANOVA) was used, followed by Fisher's test (F), to compare the means. Difference was considered significant at $p < 0.05$.

3. Results

3.1. Moisture Content

Moisture content is an important index of food stability [33]. The moisture content of okra leaves and seeds ranged from 5.7 to 6.16%. The moisture content of leaves and seeds studied was within the acceptable range of 0–13% [34]. However, the moisture content of fruit samples was 88.14%. The results of seed moisture contents in the present work

were similar to the results of the sesame seed study (6.21 ± 2.41%) reported by Christian Ebere [35]. Our results showed a lower moisture content of leaves (9.77 ± 0.00%) compared to that in the findings of Williams et al. [36]. The moisture content of okra fruit samples was found to be 88.14 ± 0.89%, which is very close to the mean value of the moisture contents reported by Polash et al. [37], which ranged from 88.77 ± 0.71 to 90.16 ± 1.

3.2. Total Phenolic Content

The total phenolic content of okra leaves, fruits and seeds are shown in Table 1. The results confirmed that all parts of *Abelmoschus esculentus* L. showed high concentrations of phenolic compounds in aqueous and ethanolic extracts, but the ethanolic solvent was more efficient than was the aqueous one in extracting phenolic compounds. The total phenolic content values ranged from 17.14 ± 0.42 mg GAE/g DW (aqueous seed extract) to 162.46 ± 4.48 mg GAE/g DW (ethanolic leaf extract). However, it is clear from the data presented in Table 1 that the total phenolic content was significantly ($p < 0.05$) higher in the okra leaf extracts. On average, the leaf extract accumulated two- and three-fold higher levels of phenolic compounds compared to those accumulated by the fruit and seed aqueous extracts, respectively. At the same time, the total phenolic content was two to four times higher in ethanolic extracts than that in aqueous extracts, which is in agreement with the findings of Liao et al. [38]. As shown in Table 2, the total phenolic content of microcapsules (TPCC) was lower than the phenolic content of uncoated phenolic powder. The total phenolic content of microcapsules ranged from 10.66 mg GAE/g (microcapsules of aqueous seed extract) to 17.47 mg GAE/g (microcapsules of ethanolic leaf extract). These results are consistent with those of the study by Saikia et al. [39], which highlighted a four-fold decrease in the phenolic content of orange powder after spray drying.

Table 1. Moisture content of dried okra leaves, fruits and seeds.

Okra Organ	Moisture Content (%)
Leaves	5.7 ± 1.54
Fruits	88.14 ± 0.89
Seeds	6.16 ± 0.87

Table 2. Yield, total phenolic content, TPC, TPCC and SPC values of okra leaves, fruits and seeds.

Samples	Yield (%)	TPC (mg GAE/g DW)	TPCC (mg GAE/g DW)	SPC (mg GAE/g DW)
Leaves (H_2O)	16.68 ± 0.492 ^{d}	46.55 ± 0.49 ^{c}	12.22 ± 0.16 ^{d}	3.26 ± 0.14 ^{a}
Fruits (H_2O)	21.22 ± 0.228 ^{b}	26.16 ± 0.21 ^{d}	13.04 ± 0.15 ^{cd}	2.66 ± 0.12 ^{bc}
Seeds (H_2O)	18.46 ± 0.104 ^{c}	16.86 ± 0.42 ^{e}	10.66 ± 0.47 ^{e}	1.86 ± 0.15 ^{d}
Leaves (EtOH)	11.15 ± 0.232 ^{e}	162.46 ± 4.48 ^{a}	17.47 ± 0.84 ^{a}	3.20 ± 0.1 ^{ab}
Fruits (EtOH)	10.30 ± 0.197 ^{f}	41.26 ± 0.99 ^{c}	15.63 ± 0.72 ^{b}	2.41 ± 0.42 ^{cd}
Seeds (EtOH)	23.94 ± 0.466 ^{a}	85.54 ± 2.05 ^{b}	14.28 ± 0.52 ^{bc}	1.00 ± 0.14 ^{e}

Note: H_2O: aqueous extract; EtOH: ethanolic extract. Data are shown as means ± SD ($n = 3$). The different lower-case letters indicate significantly different values ($p < 0.05$).

Furthermore, the change in TPC during the spray drying process can be related to the plant part and the extraction solvent. Since phenolic components are susceptible to external factors, many studies have reported their relationship with a decrease in total phenolic content in spray-dried microencapsulated powders [40–42]. This decrease can also be explained by the homogenization step during the encapsulation process. The external mechanical force of the mixture can break the interface between the phenolic powder and the coating material, which can cause mechanical damage to the phenolic compounds [31]. In addition, the chemical conformation of phenolic compounds change during the spray drying process, which can make them more soluble and decrease the TPC [39].

3.3. Encapsulation Efficiency

Encapsulation efficiency (EE) is one of the crucial indicators of the efficiency of the microencapsulation process in properly encapsulating the core material. It represents the percentage of compounds successfully encapsulated in the capsules. The effects of different plant parts and extraction solvents on encapsulation efficiency are shown in Figure 1. As can be seen in Figure 1, significant differences were observed between the encapsulation efficiencies of the okra extracts ($p < 0.05$). The EE ranged from 73.84 ± 0.10% (ethanolic leaf extract) to 90.6 ± 0.14% (ethanolic seed extract). Other previous works studying spray drying have also shown similar encapsulation efficiencies [43,44].

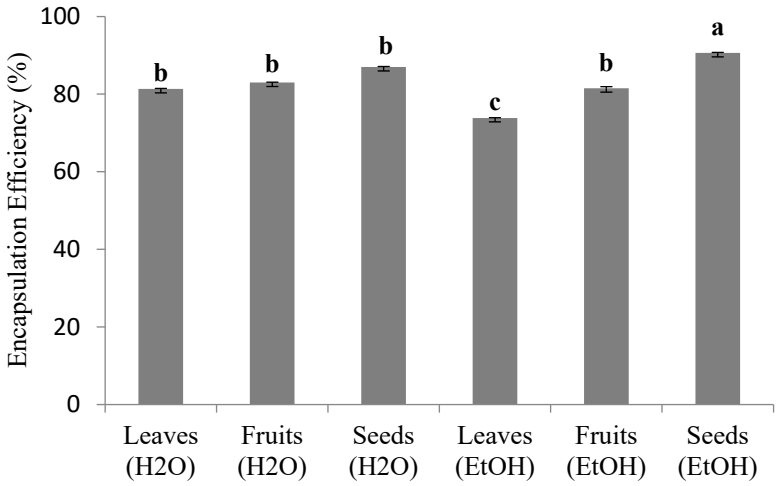


Figure 1. Encapsulation efficiency (EE) values of okra extracts. Data are shown as means and standard deviations ($n = 3$). The different lower-case letters indicate significantly different values ($p < 0.05$).

The average EE for all phenolic compounds analyzed was 82.01 ± 1.48%. Similarly, Davidov-Pardo et al. [45] reported an EE of 82% for a spray dried commercial grape seed polyphenol extract using MD as a coating material. However, a lower EE of 55 to 79% was found for encapsulated phenolic extracts from white and red wine [46]. Other authors have shown that the encapsulation efficiency is influenced by several factors, such as the technique used, the solubility of the matrix in organic solvents and the solubility of the organic solvent in water [47]. In addition, the type of coating material used and the ratio between the core material and the coating material has a strong influence on the EE. Maltodextrin has been reported to improve encapsulation efficiency due to its high solubility in water and low viscosity values even at high concentrations [21]. In addition, pectin is well suited as both a coating and gelling material [48]. Our results are in conformity with those of Sansone et al. [49] that proved the efficiency of use of a 10:1 maltodextrin/pectin weight ratio (11% *w*/*v*) matrix to encapsulate polyphenols. The MD/P mixture is capable to mask the disagreeable smell of the extracts. Homogeneity with this mixture facilitates product solubility in water. The results confirmed that spray drying could be a feasible technique to improve the quality of phenolic compounds sensitive to high-temperature [50].

3.4. Anti-Oxidant Activity

The anti-oxidant activity of non-encapsulated and spray-dried samples was determined by DPPH radical scavenging activity as shown in Figure 2. The non-encapsulated samples revealed significantly higher anti-oxidant activity compared to the encapsulated samples. In addition, the data expressed a significant difference ($p < 0.05$) in the anti-oxidant activity of uncoated phenolic powder ranging from 24.63% (aqueous seed extract) to 75.65% (ethanolic leaf extract) and coated phenolic powder ranging from 7.58% (aqueous fruit extract) to 30.63% (ethanolic fruit extract). Our data were in agreement with Simon-Brown

et al. [51], who highlighted that microencapsulation of ginger extract by spray drying technology also reduced phenolic compounds and anti-oxidant activity.

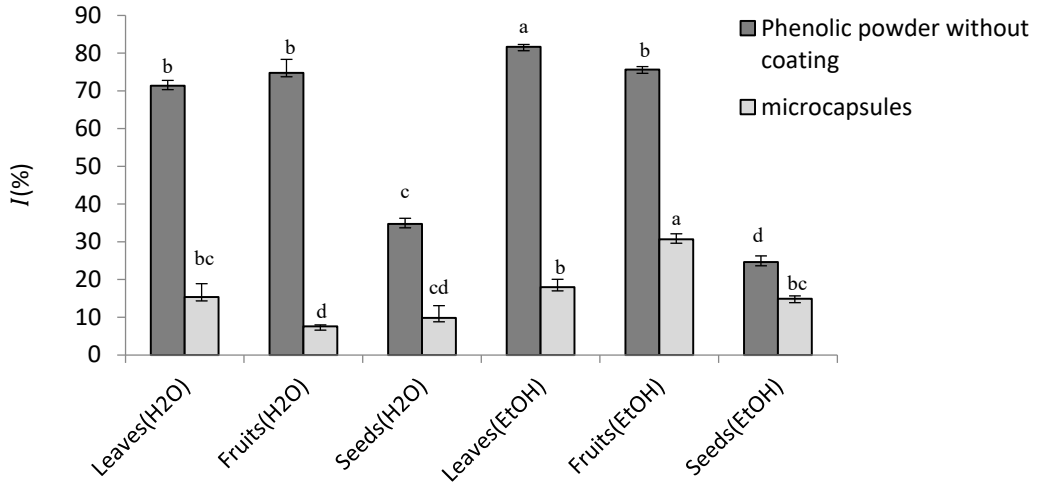


Figure 2. Anti-oxidant activity values of non-encapsulated and encapsulated phenolic powder. Data are shown as means ± standard deviation ($n = 3$). The different lower-case letters indicate significantly different values ($p < 0.05$).

The radical scavenging activity of the different parts of the okra extract was reduced by 45.80% compared to that of the encapsulated powder. This is in agreement with the results of Yang et al. [52], who reported a 65% reduction in anti-oxidant activity under heating between 65 and 75 °C. Thus, the increase in inlet temperature accelerated the loss of anti-oxidant activity in the microcapsules. Similarly, the anti-oxidant activities of spray-dried okra fruit, leaf and seed extracts were affected by the spray drying temperature, as during the encapsulation process, anti-oxidants are degraded by extrinsic factors such as temperature changes, light and oxygen [53]. Our results were also consistent with those of previous studies that reported a correlation between important anti-oxidant effects and the presence of phenolic compounds sensitive to high temperatures [54].

3.5. Particle Size of the Microcapsules

The size of the microcapsules varied between 7.98 and 13.36 µm in all experiments, except for encapsulated ethanolic seed extracts, which had a larger size of 60.96 µm (Table 3). However, the size variation of microcapsule powders is a particular characteristic of spray-dried microcapsules [55]. According to Che Man et al. [56], the particle diameter of microcapsule products obtained via the spray drying technique ranged from 1 to 15 µm. Our data were generally similar to those expressed in the literature, where the droplet size of the capsules ranged from 2 to 10 µm in diameter [31]. These characteristics of microcapsules have been reported in previous works, such as a study by Sarabandi et al. [57], who highlighted a particle size greater than 50 µm for the microencapsulation of peel extract. The molecular structure of the carrier agents explains the differences in microcapsules size [58]. Probably due to its higher emulsion viscosity, okra seed showed a larger size. Sansone et al. [49] suggested that the presence of pectin has a significant effect on particle size. Also, according to Favaro-Trindade et al. [59], capsules obtained via complex coacervation can have particle sizes ranging from 1 to 500 µm. On the other hand, a relationship was estimated between particle size distribution and span values, where a span with lower values indicates a more homogeneous particle size distribution [60]. In the same context, it has been reported in the literature that higher span values indicate a wider range of particle size distribution [61]. Subsequently, the smallest span values were detected for the spray-dried aqueous leaves and seeds, with span values below 2. Smaller diameters were also found for these samples.

Table 3. Particle size of okra microcapsules.

Microcapsules	D [4,3] (μm)	Span
Leaves (EtOH)	11.97 ± 1.58	1.94 ± 0.05
Fruits (EtOH)	8.82 ± 0.07	1.95 ± 0.01
Seeds (EtOH)	60.96 ± 102.33	22.35 ± 45.05
Leaves (H_2O)	7.98 ± 0.12	1.76 ± 0.009
Fruits (H_2O)	12.36 ± 0.54	1.93 ± 0.02
Seeds (H_2O)	9.09 ± 0.16	1.76 ± 0.003

Furthermore, the microcapsules obtained in our study were smaller than 100 μm, which allowed them to function as effective delivery systems and may simplify their utilization in food products [62]. In particular, the release rate of bioactive molecules increased as the size of the microcapsules was reduced [63]. Our findings are in line with the results of previous studies, which reported that the size of microcapsules used for food applications should be less than 100 μm to avoid affecting the oral sensation (i.e., grittiness) from the product [64]. In other ways, the small size of the capsules makes it possible to control the continuous release of the encapsulated molecules [65]. Moreover, more the microcapsules means decreased particle size and encapsulated core distribution becoming more narrow, which maintains product consistency.

3.6. Scanning Electron Microscopy of Microcapsules

Morphological analysis was used to observe the surface morphology in addition to the internal and external structure of the microcapsules (Figure 3). Analyzing the micrographs, the most common morphology of microcapsules was hemispherical with a dented surface and a continuous wall. We observed two different types of microcapsule morphologies: particles with a smooth and rounded surface (the aqueous fruit extract) and particles with a concave, collapsed and irregular surface (the rest of the extracts). In addition, in the current study, the micrographs mainly showed outer surfaces without any obvious cracks on all the particles and therefore did not lead to rupture, which is essential for the better protection of the encapsulated bioactive molecule [66]. Furthermore, the observed microcapsules without wrinkles illustrated the heat resistance and good protection of the selected core materials [67]. The wrinkling may have been due to the drying and cooling mechanism of the microcapsules in the spray dryer [51]. Similarly, Sarabandi et al. [68] mentioned that several circumstances can influence the surface morphology and particle size of spray-dried microparticles such as inlet air temperature. Accordingly, rigid microcapsules and porous surfaces were obtained as a result of high inlet air temperatures between 173 and 200 °C [69]. It is interesting to highlight that samples tend to waste their moisture faster when the drying temperature of microencapsulation is high, resulting in the efficiency of the encapsulation technique and the entrapment of biomolecules within the core [70]. Similarly, previous studies have shown similar characteristics of spray-dried Tucuma (*Astrocaryum vulgare* Mart.) co-products using maltodextrin [66] and spray-dried powder capsules of methanolic extracts of chipilin (*Crotalaria longirostrata*) leaves encapsulated with different encapsulating agents [71]. Microcapsules of ethanolic extracts looked more compressed compared to microcapsules of aqueous extracts. In addition, Ferreira et al. [66] confirmed that some microparticles can be seen with a continuous surface and others with a wrinkled surface. The microstructure of dried encapsulated extracts mainly depends on the type of encapsulation and the drying process [57]. Within this context, Ruiz-Canizales et al. [72] found the structure of maltodextrin-coated microencapsulates of blue maize phenolics to be spherical with pronounced depressions. According to the observations of Robert et al. [73], the irregular structure of the microcapsules increases their solubility properties in a food matrix to enhance the functional properties of the food.

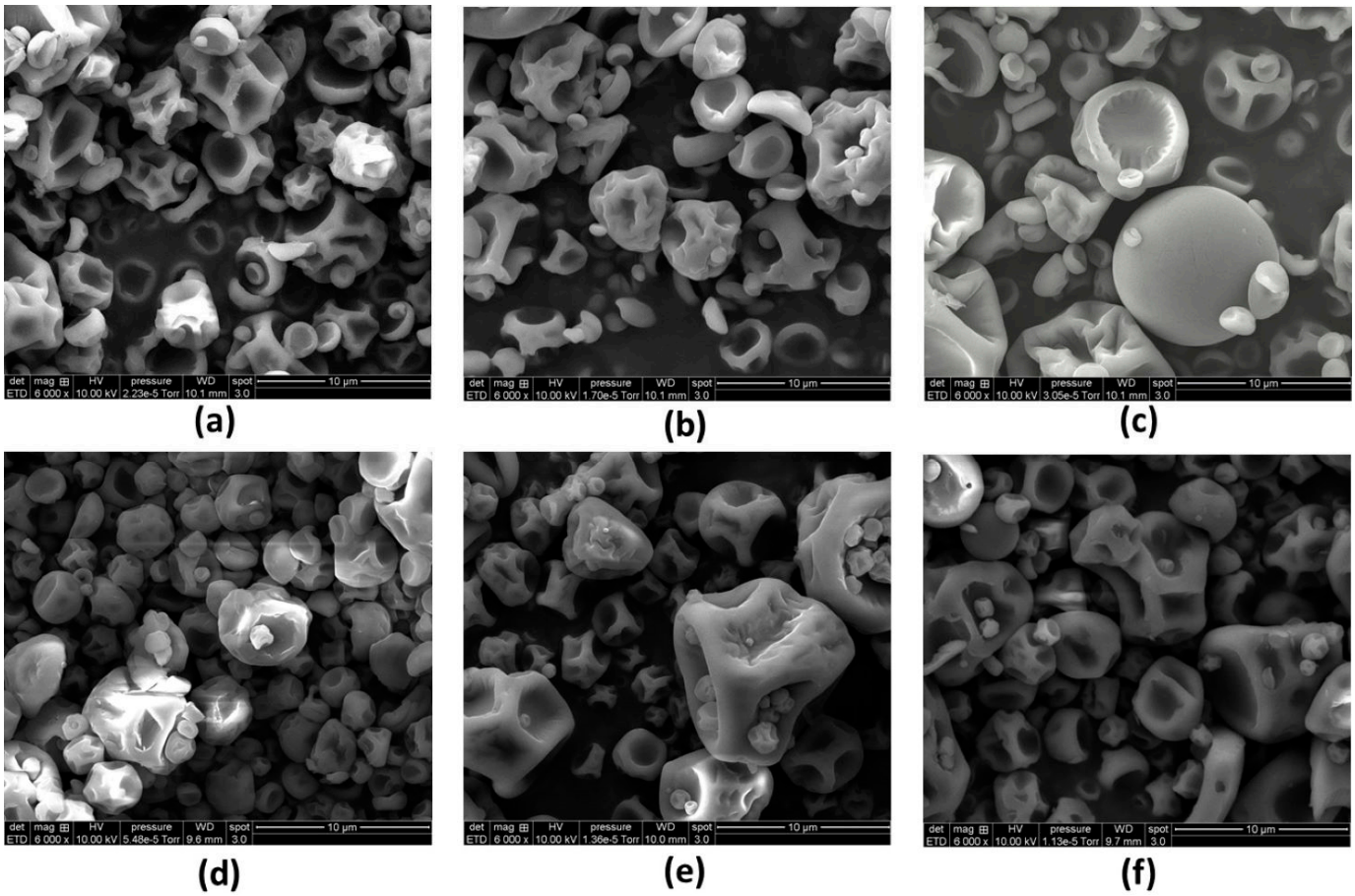


Figure 3. SEM micrographs of the microcapsules: (**a**) aqueous seed extract, (**b**) aqueous leaf extract, (**c**) aqueous fruit extract, (**d**) ethanolic seed extract, (**e**) ethanolic leaf extract and (**f**) ethanolic fruit extract.

3.7. Antibacterial Activity

In the present study, phenolic compounds from *Abelmoschus esculentus* L. before and after encapsulation were evaluated for their antibacterial activity against two Gram-positive (*Listeria innocua* and *Brochotrix thermosphacta*) and two Gram-negative (*Escherichia coli* and *Salmonella enterica*) bacteria. Figure 4 shows the inhibition zones for unencapsulated and encapsulated samples. Interestingly, the data showed clear zones of inhibition against the two Gram-positive bacteria, but no zones of inhibition against the Gram-negative bacteria. This is in line with the hypothesis that Gram-positive bacteria are more sensitive to plant phytochemicals [74]. These findings can be interpreted according to the differences in the cell composition of Gram-positive and Gram-negative bacteria, which lead to differences in the resistance of both bacteria to different treatments due to the protection of the bacterial cell wall [75].

The growth of *Listeria innocua* and *Brochotrix thermosphacta* was inhibited by both aqueous and ethanolic microcapsules. Ethanolic microcapsules tended to be more effective in limiting bacterial growth. On the other hand, both ethanolic and aqueous seed microcapsules tended to be more effective in inhibiting the bacterial proliferation of *Listeria innoua*, with a significant inhibition zone of 38 and 30 mm, respectively. So far, Ennadir et al. [76] have suggested that bacteria are more sensitive to organic seed extracts than to aqueous extracts of two therapeutic herbs (*Nigella sativa* L. and *Foeniculum vulgare Mill*). Furthermore, previous studies showed that ethanolic seed extract exhibited the maximum inhibitory effect compared to the aqueous extract [77]. On the other hand, Petropoulos et al. [78] and Oloketuyi [77] highlighted the significant antimicrobial activities of okra seed extract, especially against *Listeria monocytogenes*. Thus, *Abelmoschus esculentus* L. seeds could

be considered an innovative end-use products for the food and pharmaceutical industries, in particular for functional foods with antimicrobial and anti-oxidant effects. Furthermore, *Listeria innocua* and *Brochotrix thermosphacta* were more sensitive to microencapsulated than nonencapsulated okra phenolic compounds. These results are in agreement with those of Matouskova et al. [79], who highlighted that encapsulated particles were more active against several bacteria stains and more stable and more effective than were the uncoated ones in the case of long-term storage.

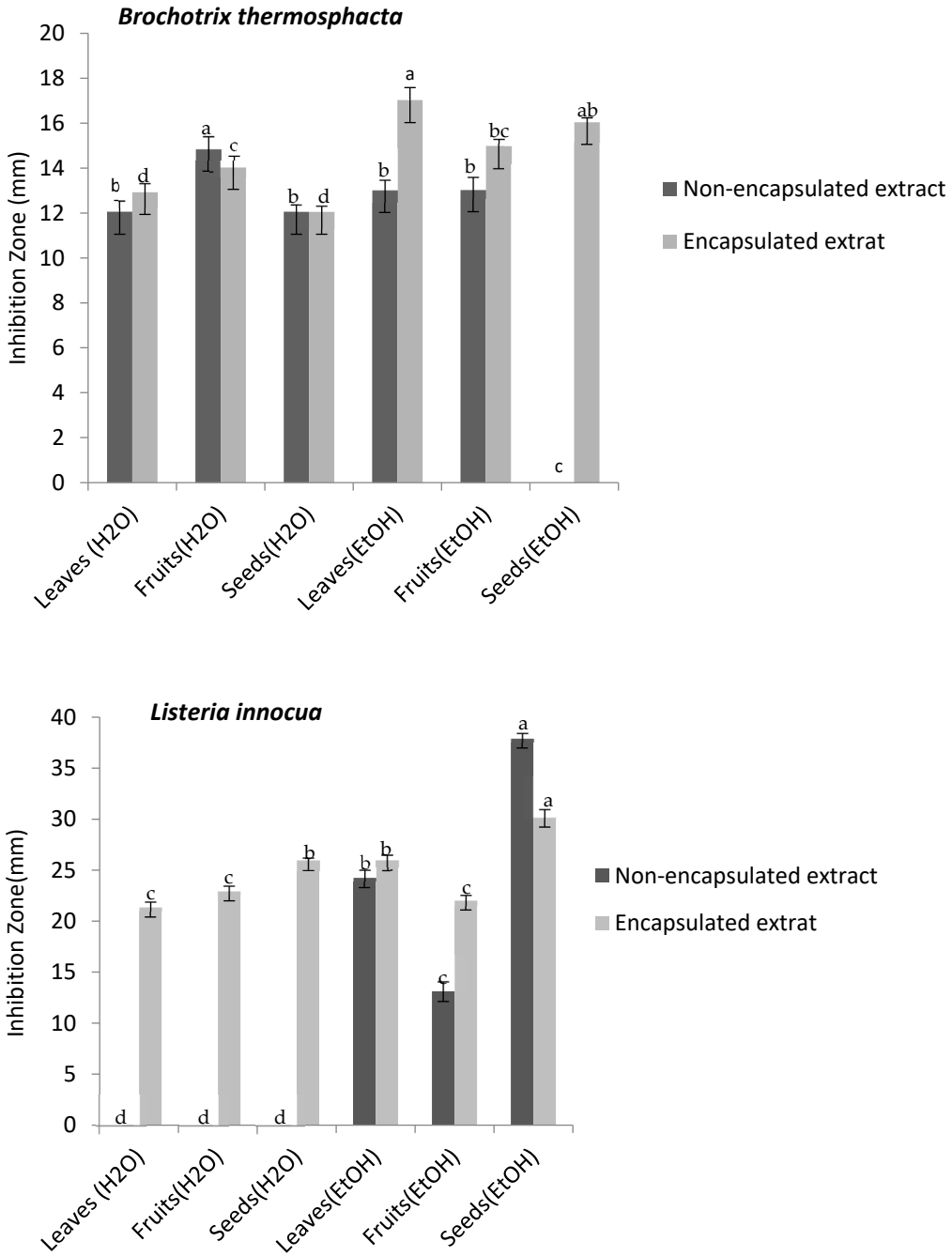


Figure 4. Inhibition zones of non-encapsulated and encapsulated phenolic compounds from okra leaves, fruits and seeds. Data are shown as means ± standard deviation (n = 3). The different lower-case letters indicate significantly different values ($p < 0.05$).

4. Discussion

All okra extracts have been shown to be an important source of phenolic compounds and may be useful as natural anti-oxidants in food and pharmaceutical products. Fur-

thermore, the observed decrease in total phenolic content and anti-oxidant effects of encapsulated *Abelmoschus esculentus* L. extracts was in agreement with those reported in the literature [80]. Additionally, the change in TPC during the spray drying process can be related to the plant part and the extraction solvent. Moreover, similar results were reported by Suhag and Nanda [81] and Singh et al. [82], highlighting the efficiency of maltodextrin and pectin in protecting the phenolic molecules of spray-dried honey and jamun pulp powders, respectively.

Similarly, encapsulation efficiency is one of the important factors influencing the amount of biomolecules remaining on the surface of encapsulated microparticles [83]. This demonstrated the ability of the spray drying encapsulation process technology to reduce the oxidative breakdown of active particles [84]. Our data showed an important encapsulation efficiency value (82.01 $\pm$ 1.48%) which highlighted the ability of the maltodextrin/pectin mixture to protect the core materials, and this is one of the most important parameters to determine the sustainability of the encapsulated compounds [85]. In the same context, a previous study reported that microcapsules with a core to shell ratio of 1:10 had high encapsulation efficiency and anti-oxidant activity, which was explained by efficient mixing due to the low viscosity of the solution [86]. Thus, similar data have demonstrated the close relationship between the increase in encapsulation efficiency of microencapsulated phenolic compounds and the mixture of a maltodextrin/pectin matrix as an alternative to achieve effective encapsulation [49,87]. Furthermore, the size variation of okra microcapsules is an exclusive feature of microcapsules derived from the spray drying process [55]. The results showed that the encapsulated phenolic compounds of okra aqueous and ethanolic extracts were of a size between 7.98 and 60.96 µm. Furthermore, previous studies by Vaucher et al. [88] also reported similar results regarding spray-dried particle sizes ranging from 2 to 120 µm for microencapsulated fish oil.

In addition to the important anti-oxidant activities of okra capsules, encapsulated extracts of *Abelmoschus esculentus* L. have significant bactericidal activity. Both aqueous and ethanolic microcapsules are able to inhibit the proliferation of *Listeria innocua* and *Brochotrix thermosphacta*. However, our findings are in accordance with those previously published by Fancello et al. [89], who showed a higher sensitivity of Gram-positive bacteria than that of Gram-negative bacteria against *Citrus limon* microparticles. Thus, the encapsulation of plant extract particles increased their antimicrobial activity. This consequence could be due to the higher stability of the active molecules encapsulated in the polymeric capsule. Otherwise, we demonstrated the potential of using non-encapsulated and encapsulated okra extracts as antibacterial biomolecules with possible applications in the food and pharmaceutical industries. As a conclusion, our results clearly highlight the resistance of the encapsulation agents and their stability under mechanical constraints during the spray drying procedure. As a consequence, the good encapsulation efficiency of the Tunisian phenolic extracts was demonstrated.

5. Conclusions

The current study has shown that different parts of Tunisian okra contain an important amount of bioactive polyphenolic compounds and presented their considerable anti-oxidant and antibacterial effects. In this work, the microencapsulation of aqueous and ethanolic extracts of *Abelmoschus esculentus* L. leaves, fruits and seeds was carried out using a maltodextrin/pectin mixture by using spray-drying. In addition, the ethanolic *Abelmoschus esculentus* L. fruit and leaf microparticles demonstrated a valuable anti-oxidant effect, suggesting their possible use as a multi-disciplinary dietary food additive or supplement. This study demonstrated that okra microparticles exhibited important antibacterial activity against *Brochotrixt hermosphacta* and *Listeria innocua*. In conclusion, this study responds to the growing interest of the food industry and consumers in using natural biomolecules that can extend the shelf life of perishable foods.

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