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Effect of different drying methods on phytochemical content and amino acid and fatty acid profiles of the green seaweed, *Ulva* spp.

Elsa Uribe[1,2] · Antonio Vega-Gálvez[1] · Vivian García[1] · Alexis Pastén[1] · Jéssica López[3] · Gabriela Goñi[4,5]

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Abstract
Green seaweeds are a potential source of proteins, minerals, fatty acids, and essential amino acids, and also often contain bioactive compounds with antioxidant activity. They have the potential to be a source of functional and nutraceutical ingredients. However, their elevated water content shortens shelf life; thus, a preservation method should be employed, such as drying. In the present article, Chilean green seaweed (*Ulva* spp.) was characterized and the effect of different drying methods (freeze-, vacuum-, solar-, and convective drying) on the quality of dried algae as functional ingredient, along with a description of the drying parameters for each method was evaluated. Proximate composition of fresh *Ulva* spp. indicated that, other than water, ash, protein, and crude fiber are the main constituents. *Ulva* samples also had a high amount of total dietary fiber (with an IFD/SFD ~ 1.5). The isotherm curve presented the typical type II sigmoid shape and the BET model gave the best fitting. There was a significant effect of drying method on proximate composition of dried *Ulva* and the convective drying the method that showed higher values for almost all parameters, except fat content. Color was not affected by drying and the typical green color was present in all samples. Total flavonoid content (TFC), total carotenoids and antioxidant capacity (DPPH and ORAC) were also higher in convective drying. In addition, other minor components with nutritional value were identified, such as essential polyunsaturated fatty acids (PUFAs with a ω3/ω6 ratio of 1:1) and amino acids. Among the different drying methods applied, convective drying (70 °C, 120 min) better retained the physicochemical parameters and antioxidant capacity of *Ulva* spp.

Keywords Clorophyta · Macroalgae · Convective drying · Solar drying · Vacuum drying · Antioxidant capacity · Dietary fiber

Introduction

Amid growing concern by consumers for healthy, nutritious foods with additional health-promoting functions, the food industry is driving the use of new technologies to produce functional foods (Chan and Matanjun 2017). Seaweeds represent a renewable natural resource with potential use as functional ingredient due to their valuable physicochemical composition (Ortiz et al. 2006; Holdt and Kraan 2011). Seaweed composition is not as well known as land plants (Chan and Matanjun 2017) and many factors have a strong impact on their chemical composition, such as species, geographical localization, maturity, environmental conditions, etc. (Peña-Rodríguez et al. 2011), which make generalizations very inadequate. Green seaweeds of the genus *Ulva* (Chlorophyta) are distributed worldwide (Peña-Rodríguez et al. 2011), and several of them are common along the Chilean coasts (Ortiz et al. 2009). They represent an important biomass in eutrophicated coastal areas causing economic and ecological problems (Yaich et al. 2015), but at the same time, their interesting nutritional composition makes their commercial exploitation attractive to produce functional food ingredients (Peña-Rodríguez et al. 2011; Postma et al. 2018). Moreover, *Ulva* is a very adaptable genus that can readily adapt its metabolism to different environmental conditions

✉ Elsa Uribe
muribe@userena.cl

[1] Food Engineering Department, Universidad de La Serena, Av. Raúl Bitrán 1305, La Serena, Chile

[2] Instituto de Investigación Multidisciplinar en Ciencia y Tecnología, Universidad de La Serena, Av. Raúl Bitrán 1305, La Serena, Chile

[3] Escuela de Alimentos, Pontificia Universidad Católica de Valparaíso, Waddington 716, Playa Ancha, 2360100 Valparaíso, Chile

[4] Grupo de Investigación en Ingeniería de Alimentos (GIIA). Departamento de Ingeniería Química, Facultad de Ingeniería, Universidad Nacional de Mar del Plata, Av. Juan B. Justo 4302, Mar del Plata, Argentina

[5] Consejo Nacional de Investigaciones Científicas y Técnicas (CONICET), Buenos Aires, Argentina

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(McCauley et al. 2016). These adaptive characteristics of *Ulva* would mean that it can be easy to cultivate (Peña-Rodríguez et al. 2011; Mata et al. 2016).

Ulva spp. contain up to 44% of proteins based on the algae dry weight (Rioux et al. 2017), a complete profile of amino acids (Ortiz et al. 2006), besides valuable polyunsaturated fatty acids (PUFAs) with a favorable ω6/ω3 ratio (McCauley et al. 2016). They are also excellent sources of polysaccharides, with high dietary fiber content (Yaich et al. 2015) and presence range of minerals, vitamins, and trace elements (Yu-Qing et al. 2016; Chen et al. 2017; Rioux et al. 2017; Wells et al. 2017). Studies on the biological functionalities of isolated metabolites from *Ulva* species have revealed numerous health-promoting effects, including anti-oxidative, anti-inflammatory, anti-microbial, anti-viral, and anti-cancer effects (Yu-Qing et al. 2016; Li et al. 2018).

However, to obtain a product suitable for industrial processing, it is necessary to apply a preservation technique, such as drying. The drying operation is responsible to removing a large amount of water present in the seaweed, which eventually decreases the water activity and retards the growth of microorganisms and reduces the weight and volume which has a positive impact in cost of storage and transportation (Tello-Ireland et al. 2011). Even though drying conditions may significantly affect the nutritional, functional, and biological properties of seaweed, their bioactivity and nutritional value will depend mainly on the drying methods used (Gupta et al. 2011; Stévant et al. 2018). Fresh seaweeds contain a large amount of water (75–85%) and are usually dried before being used in nutritional studies or industrial processing (Neoh et al. 2016). Currently, several drying technologies are used for seaweeds (Ling et al. 2015). Sun drying is the common and cheapest method. However, product quality is affected by weather and microbial attack, contaminations by dust, insects and birds, the difficulty of process control, and bad odor (Fudholi et al. 2014). This problem could be eliminated if seaweeds are dried under isolated controlled conditions using, for instance, convective-, vacuum-, or freeze-dryers. Convective air-drying is widely used for its low equipment and operation cost, although the maintenance of food quality attributes through this process has presented some serious problems (Tello-Ireland et al. 2011). Conversely, vacuum drying can help to enhance dried product quality and nutritive value of seaweeds (Uribe et al. 2018), leading to reduced drying times by lowering the pressure during the process and absence of oxygen. Freeze-drying is generally considered as the best method for production of high-quality dried products. However, disadvantages include high production costs, high energy consumptions, and low throughputs (Hsu et al. 2003). In this context, Chan et al. (1997) found differences in amino acid and fatty acid profiles of the brown seaweed *Sargassum hemiphyllum* after being freeze-, sun-, and oven-dried. Wong and Cheung (2001) showed that, on one hand, freeze-drying was the most appropriate drying method in retaining the nutritional composition of three *Sargassum* species, while, on the other hand, oven-drying was better than freeze-drying for the extractability and in vitro digestibility of proteins isolated from these seaweeds. Ling et al. (2015) compared seven different drying techniques and their effect on the phytochemical content and antioxidant activity of a red seaweed (*Kappaphycus alvarezii*). They found that oven-drying at 40 °C showed the highest values of phytochemical compounds and displayed better scavenging and reducing ability. In the same species, Neoh et al. (2016) also found that oven-drying (at 60 °C) provided the highest antioxidant activity values, but vacuum-dried seaweed possessed better phenol and flavonoid total contents. However, little information is found on the effect of drying in *Ulva* spp.; hence, investigation of the effects of different drying methods on them is necessary.

The aim of the present study is to evaluate the effect of different drying methods (freeze-, vacuum-, solar-, and convective drying) on the nutritional quality of *Ulva* spp. as a potential functional ingredient. Therefore, bioactive compounds, antioxidant capacity, and amino acid and fatty acid profiles were measured for each dried sample. In addition, a characterization of fresh *Ulva* spp. was performed.

Materials and methods

Seaweed sampling

Vegetative thalli of *Ulva* spp. (*Ulva lactuca* Linnaeus and *Ulva rigida* Agardh; Oróstica et al. 2017) were collected at up to 4 m depth from Guayacan beach, Region of Coquimbo, Chile, in April 2017. The seaweeds were rinsed and maintained in 1000-L raceway tanks (Ocean Teach S. A, Chile), which received a constant flow of filtered sea water at a rate of 150 L h^{-1}. Upon arrival at the laboratory, the algal samples were washed again with distilled water and subjected to visual inspection by size, homogenous color, and absence of mechanical damage. A portion of the fresh *Ulva* spp. was used in the proximate composition analyses following standard methodologies, while the rest of the fresh seaweed was used for the different drying experiments.

Drying method conditions

Ulva spp. were dried by different drying methods: (i) lyophilization (freeze-drying, FD) using a freeze-dryer VirTis Wizard 2.0 (Advantage Plus, USA), (ii) vacuum drying (VD) using a vacuum oven (Memmert, model VO 400, Germany), (iii) solar drying (SD), and (iv) convective drying (CD) using a solar and convective dryer, respectively, both designed and built at the Department of Food Engineering,

Universidad de La Serena, Chile. FD samples (540 g) were first frozen at − 80 °C for 24 h, and then quickly placed into the freeze-dryer (− 50 °C, 0.027 KPa, 68 h). VD samples (250 g) were placed in a single layer on a stainless steel tray at a density of 2.07 kg m^{-2} inside the vacuum dryer (70 °C, 15 kPa). The SD process was performed in a solar dryer with an integrated flat-plate collector which used a copper plate to absorb the incident solar radiation and a glass sheet as the transparent cover. SD samples (1000 g) were spread on a stainless steel tray at a density of 2.06 kg m^{-2} (approximately at 50 °C, 30–40% humidity, about 8 h of daylight). CD samples (400 g) were placed in the drying chamber in a single layer at a load density of 4.19 kg m^{-2} (hot air temperature = 70 °C, air flow rate = 2.0 m $s^{-1)}$. All drying methods were carried out until constant weight, except for FD where samples were kept in the drying chamber for 68 h. Once the samples were dried, they were ground using a basic analytical mill (IKA A-11, USA), sieved with a stainless steel sieve #35 of 500 μm mesh (U.S. Standard Sieve Series, Dual Manufacturing Co., USA), and then stored in sealed plastic bags at 5 °C until tested.

Desorption isotherm model

Desorption isotherm for fresh *Ulva* spp. was determined at 50 °C according to the method recommended by Spiess and Wolf (1993). The fit of experimental equilibrium moisture content (X_{we}) and water activity (a_w) were performed using the BET model (Eq. (1)) where X_m, the monolayer moisture content (g water g^{-1} DM) and C (dimensionless), are the BET model parameters and a_w (dimensionless) is the water activity. This equation has been widely used to describe the equilibrium moisture content of seaweeds (Lemus et al. 2008; Moreira et al. 2016a, b):

$$X_{we} = \left[\frac{X_m C\, a_w}{\left((1-a_w)(1+(C-1)a_w)\right.}\right] \quad (1)$$

Drying characteristics and drying rate curve

The characteristic curve of drying allows us to study the effects of different drying conditions on *Ulva* spp. drying kinetics. First, moisture content data was converted to moisture ratio expression (MR) that relates the sample moisture content in real time (X_{wt}) to the initial moisture content (X_{wo}) and the equilibrium moisture content (X_{we}). The average MR was calculated using Eq. (2):

$$MR = \frac{X_{wt}-X_{we}}{X_{w0}-X_{we}} \quad (2)$$

In addition, drying rate (DR) refers to moisture loss per unit time, which can be calculated according to Eq. (3) where t_1 and t_2 are the drying time, min, Xw_{t1} and Xw_{t2} are the moisture contents on dry basis, g g^{-1}, at time t_1 and t_2, respectively:

$$DR = \frac{X_{wt_2}-X_{wt_1}}{t_2-t_1} \quad (3)$$

Phytochemical properties of *Ulva* spp.

Determination of proximate composition

Ulva spp. moisture content, crude protein content using a conversion factor of 5 (Angell et al. 2016), fat, crude fiber, and ash were determined according to AOAC methodology (AOAC N° 934.06; AOAC N° 960.52; AOAC N° 960.39; AOAC N° 962.09; AOAC N° 923.03, respectively) on fresh and dried samples. Water activity of the *Ulva* spp. samples was measured with an AQUA LAB equipment (4 TE, Pullman, WA, USA) at 25 °C. Results are expressed in dry matter (DM) basis and all measurements were performed in triplicate, and water activity is dimensionless.

Determination of dietary fiber content

Soluble and insoluble dietary fiber (SDF and IDF, respectively) content in *Ulva* spp. samples were determined by the gravimetric enzymatic method (AOAC N° 991.43). A Total Dietary Fiber Assay Kit (TDF100A; Sigma-Aldrich, USA), an Enzymatic Digestion Unit, and a Filtration System (VELP Scientifica, GDE-CSF6, Italy) were used. Total dietary fiber (TDF) was calculated as the sum of SDF and IDF, express as gram $(100\ g\ DM)^{-1}$.

Measurement of color

Superficial color of *Ulva* spp. samples was measured with a colorimeter (HunterLab, MiniScan XE Plus, USA) using the CIE Lab coordinates, L^* (lightness), a^* (redness/greenness), and b^* (yellowness/blueness). In each sample, five replicates were measured and the average is presented. Total color difference (ΔE) was calculated according to Eq. (4), where L_0, a_0, and $b_0$corresponded to fresh *Ulva* spp. Polar coordinates of color Chroma (C*) and hue angle (h^*) were calculated according to Eq. (5) and Eq. (6), respectively:

$$\Delta E = \sqrt{\left(a^*-a_0\right)^2 + \left(b^*-b_0\right)^2 + \left(L^*-L_0\right)^2} \quad (4)$$

$$C^* = \left(a^{*2} + b^{*2}\right) \quad (5)$$

$$h^* = tan^{-1}\left(\frac{b^*}{a^*}\right) \quad (6)$$

Bioactive compounds and antioxidant capacity

Extraction of antioxidant compounds

One gram of dried *Ulva* spp. was weighed and transferred with 50 mL of 60% methanol solution to an Erlenmeyer flask in an orbital shaker (Boeco, OS20, Germany) at 25 °C and 200 rpm. After 24 h, it was filtered through a Whatman #1 filter paper into a 250-mL round bottom flask and the solvent was completely evaporated under reduced pressure at 40 °C in a rotary evaporator (Büchi R-210, Switzerland). Dried residue was resuspended in 10 mL of 60% methanol. Samples were protected from light throughout the extraction process. These methanolic extracts were kept in dark cold.

Determination of total phenolic content

Total phenolic content (TPC) was determined using the colorimetric assay with Folin-Ciocalteau reagent as described by Uribe et al. (2018). TPC was measured in the methanolic extracts of dried *Ulva* spp. at 725 nm of absorbance and using gallic acid (Merk, Germany) as standard for the calibration curve. Results are expressed as gallic acid equivalent per 100 g of dry matter (mg GAE (100 g DM)$^{-1}$). All measurements were performed in triplicate.

Determination of total flavonoids content

Total flavonoid content (TFC) in dried *Ulva* spp. samples was carried out following the methodology proposed by Uribe et al. (2016). Catechin was used as standard in the calibration curve and results are expressed as milligrams of catechin equivalents per 100 g of dry mass (mg CE (100 g DM)$^{-1}$); all measurements were performed in triplicate.

Measurement of antioxidant activity by DPPH and ORAC methodology

The antioxidant activity of died samples of *Ulva* spp. was measured by the DPPH methodology described by Uribe et al. (2016), using synthetic antioxidant Trolox as standard for the calibration curve. Results are expressed as micromole Trolox equivalent per 100 g of dry matter (μmol TE (100 g DM)$^{-1}$); all measurements were performed in triplicate.

Antioxidant activity was also determined by the oxygen radical absorbance capacity (ORAC) on the methanolic extract of dried *Ulva* spp. obtained according to Uribe et al. (2018). Trolox was used as a standard for the calibration curve and results are expressed as micromole TE (100 g DM)$^{-1}$. All measurements were performed in triplicate.

Determination of chlorophyll content

Chlorophyll *a* (Chl*a*) and *b* (Chl*b*) content in dried samples of *Ulva* spp. was determined as described by Lichtenthaler and Wellburn (1983). One gram of ground sample was homogenized with 25 mL of acetone for 1 min. The extracts were centrifuged at 4500×*g* at 4 °C for 10 min. The absorbance of the supernatants was read at 645 and 662 nm. Chl*a* and Chl*b* were calculated according to Eq. (7) and Eq. (8), respectively. Results are expressed as micrograms of chlorophyll per gram of dry mass (μg g^{-1} DM) and all measurements were performed in triplicate:

$$\text{Chlorophyll } a = 11.75A_{662} - 2.350A_{645} \quad (7)$$

$$\text{Chlorophyll } b = 18.61A_{645} - 3.960A_{662} \quad (8)$$

Determination of total carotenoids content

Total carotenoids content (TCC) in *Ulva* spp. was determined according to Chan and Matanjun (2017). TCC was calculated following Eq. (9) where v = total volume of the extract (mL), w = sample weight (g), and $A^{1\%} = 2600$ (extinction coefficient for β-carotene extracted in hexane):

$$\text{Total carotenoid content } \left(\mu g\ g^{-1}\right) = \frac{A \times V \times 10^4}{A^{1\%} \times w} \quad (9)$$

Determination of fatty acid profiles of *Ulva* spp.

Fatty acids (FA) were determined in a gas chromatograph as methyl esters, according to European Standard NF E ISO 5509. Determinations of fatty acid profiles in *Ulva* spp. were carried out in a Perkin Elmer gas chromatograph with SGE capillary column, BPX70 bonded phase in fused silica 60 m length, 0.25 mm internal diameter and 0.25 mm film thickness with a flame ionization detector (FID), with an injector and a temperature detector at 250 °C; carrier gas was helium at 1.0 mL min^{-1} constant flow. Fatty acid content was expressed as grams per 100 g of dry matter (g (100 g DM)$^{-1}$). All samples were analyzed in triplicate.

Determination of amino acid profiles of *Ulva* spp.

The amino acid profile in dried *Ulva* spp. was determined as described by Uribe et al. (2018), using a HPLC equipped with an UV-detector and a post-column ninhydrin derivatization (Spectra-Physics spectra system pump P4000, an autosampler As1000, a UV2000 detector and PCX3100 Pickering Post Column Reactor, Pickering Laboratories, USA). Samples were buffered to pH 2.2, and norleucine was used as internal standard. Measurements were performed in triplicate.

Statistical analysis

A one-way of variance analysis (ANOVA) was performed using Statgraphics Centurion XVI (Statistical Graphics Corp., USA) to determine significant differences among the different treatments. When differences were found, comparison among means were analyzed by using the least significant difference (LSD) test with a significance level of α=0.05 and a confidence interval of 95% ($p < 0.05$). In addition, the multiple range test (MRT) was used to demonstrate the existence of homogeneous groups within each of the parameters.

Results and discussion

Proximate composition analyses of fresh *Ulva* spp.

The proximate composition and dietary fiber content of fresh *Ulva* spp. is presented in Table 1. Little information was found regarding proximal composition of fresh seaweeds, least of all about *Ulva* genus obtained in the Chilean area. Seaweeds have a variable composition depending on species, geographic habitat, water conditions, and harvest time, among other factors (Rohani-Ghadikolaei et al. 2012). However, one common characteristic of all seaweeds is the elevated water content, over 70% which indicates that they need to be consumed right after harvest or dried (Gupta et al. 2011). Concurring, moisture content from fresh *Ulva* spp. was 80.05 g (100 g)$^{-1}$, with a_w of 0.995. These results are consistent with a marine product, and reaffirm the need for dehydration to ensure quality and extend shelf life of the fresh seaweeds. In addition, ash and crude protein were the main components of fresh *Ulva.*

Table 1 The proximate composition, contents of total phenolics (TPC) and flavonoids (TFC), and antioxidant activity of fresh *Ulva* spp.

Composition	
Moisture, g (100 g)$^{-1}$	80.05 ± 0.20
Fat, g (100 g DM)$^{-1}$	2.11 ± 0.12
Ash, g (100 g DM)$^{-1}$	22.34 ± 1.11
Crude protein, g (100 g DM)$^{-1}$	10.78 ± 0.12
Crude fiber, g (100 g DM)$^{-1}$	6.27 ± 0.16
Water activity (dimensionless)	0.995 ± 0.006
Total dietary fiber, g (100 g DM)$^{-1}$	45.74 ± 1.18
Insoluble fiber, g (100 g DM)$^{-1}$	32.71 ± 2.26
Soluble fiber, g (100 g DM)$^{-1}$	13.04 ± 1.08
TPC, mg GAE (100 g DM)$^{-1}$	145.05 ± 13.58
TFC, mg CE (100 g DM)$^{-1}$	10.97 ± 0.49
DPPH, μmol TE (100 g DM)$^{-1}$	1114.0 ± 18.4
ORAC, μmol TE (100 g DM)$^{-1}$	4567.7 ± 91.3

Standard deviation was calculated on three replicates. Values are expressed as mean ± standard deviation

TPC total phenolic content, *TFC* total flavonoid content, *DPPH* 2,2-diphenyl-1-picrylhydrazyl, *ORAC* radical absorbance capacity, *GAE* gallic acid equivalents, *CE* catechin equivalents, *TE* trolox equivalents

Ash is known to be one of the main components of dried seaweeds, with values between 22 and 36% as reported by Makkar et al. (2016). Similar results were reported by Ortiz et al. (2009) for the green seaweed *Codium fragile*, Yaich et al. 2011 for *U. lactuca*, Peña-Rodríguez et al. (2011) for *Ulva clathrata* and Rohani-Ghadikolaei et al. (2012) for *Ulva intestinalis*. Seaweeds concentrate minerals from sea water, often containing 10 times the amount of minerals than land plants (Cabrita et al. 2016; Makkar et al. 2016). Therefore, the high ash content present in *Ulva* spp. could be considered as a nutritional advantage, increasing its relevance as a potential ingredient to increase mineral content in foods.

Seaweeds are often employed as a cheap protein source, especially in developing countries, due to their relatively high protein content, which can vary between 10 and 30% (Peng et al. 2015) which is in agreement with the findings of the present study (10.78 g (100 g DM)$^{-1}$). This value is lower than the one reported by Makkar et al. (2016) (18 g (100 g)$^{-1}$), but it is included in the range reported for different *Ulva* species, from 7 to 21% by Peng et al. (2015). Rohani-Ghadikolaei et al. (2012), Ortiz et al. (2006), and Yaich et al. (2011) reported 17.1, 27.2, and 8.46 g (100 g DM)$^{-1}$ for *U. lactuca*, Rohani-Ghadikolaei et al. (2012) reported 10.5 g (100 g DM)$^{-1}$ for *U. intestinalis*, and Peña-Rodríguez et al. (2011) reported 23.0 g (100 g)$^{-1}$ for *U. clathrata*. This crude protein content could be compared to other protein sources like some land plants, seeds, eggs, and grains (Ortiz et al. 2009).

Lipid content in seaweeds is usually lower than 5 g (100 g DM)$^{-1}$ (Rohani-Ghadikolaei et al. 2012), which could be considered as an advantage as for their low calorie content, along with the nutritional quality of those lipids since half of them are polyunsaturated fatty acids (Peng et al. 2015). Lipid content in fresh *Ulva* spp. was 2.11 g (100 g DM)$^{-1}$, similar to those reported by Makkar et al. (2016). These results are in agreement with Peña-Rodríguez et al. (2011) but are lower than Yaich et al. (2011) and higher than Ortiz et al. (2006). Again, variation of fat content can be explained by changes in environmental conditions, species, or extraction method employed (Ortiz et al. 2006).

Crude fiber content in fresh *Ulva* spp. was 6.27 g (100 g DM)$^{-1}$, in agreement with Makkar et al. (2016). Total dietary fiber (TDF) was 45.74 g (100 g)$^{-1}$ in fresh *Ulva* spp. This result was in accordance with previous results reported in several genus of seaweed, ranging from 32 to 71 g (100 g DM)$^{-1}$ (Chan and Matanjun 2017). Yaich et al. (2011) reported a similar value for *U. lactuca*. Peña-Rodríguez et al. (2011) reported 24.8 to 40.6 g (100 g)$^{-1}$ of TDF for cultivated *U. clathrata.* In fresh samples of *Ulva* spp., IDF represented the 71.5% of TDF, with an IDF/SDF ratio of 2.51. It is

generally stated that a suitable dietary fiber source should have an IDF/SDF ratio close to 2 (Chan and Matanjun 2017); thus, *Ulva* spp. presents the potential to be used as a functional ingredient in the food industry.

Desorption isotherm of *Ulva* spp.

The resulting desorption isotherm for *Ulva* spp. is presented in Fig. 1. It shows that the isotherm curve has the typical type II sigmoid shape, in agreement with Lemus et al. 2008 and Vega-Gálvez et al. 2008. This behavior could possibly be due to the high content of polysaccharides and proteins present in the biomass (Vega-Gálvez et al. 2008). The equilibrium moisture content increased gradually at water activity below 0.5, followed by a sharp increase above 0.85 at constant temperature (50 °C). Similar trends were observed by Lemus et al. (2008) for *Gracilaria*, Vega-Gálvez et al. (2008) for *Macrocystis pyrifera*, Moreira et al. (2016a) for *Bifurcaria bifurcata*, Moreira et al. (2016b) for *Fucus vesiculosus*, Uribe et al. (2017) for *Durvillaea antarctica*, and Uribe et al. (2018) for *Pyropia orbicularis*.

Drying kinetics are dependent on the processing conditions, and the experimental data obtained for each methodology studied. Desorption isotherms of *Ulva* spp. were modeled, finding that the BET model (Eq. (1)) was the best model for fitting the experimental desorption curves of the type II sigmoid shape, and thus presented the lowest SEE (0.00259) and χ^2 (chi-square) (0.00316) and the highest R^2 (0.954). The value of the C parameter of the BET model was 6.11 at 50 °C, which is within the ranges reported previously (2.9–23.8) for desorption isotherms of *F. vesiculosus* at 45 °C (Moreira et al. 2016b) and *B. bifurcata* at 40 and 55 °C (Moreira et al. 2016a). On the other hand, the X_m (monolayer water content) value obtained from the desorption isotherm was 0.036 g water (g DM)$^{-1}$. This value is the optimal moisture content to physical and chemical stability of seaweeds that desorb water during storage or processing (Moreira et al. 2016b). Similar values for X_m have been obtained when working with different seaweeds (Lemus et al. 2008; Moreira et al. 2016a, b). The respective equilibrium moisture content of *Ulva* spp. dried at 50 °C (solar drying) and 70 °C (convective- and vacuum drying) predicted by the BET model was 2.88 and 1.39% DM. These values were used to determine the moisture ratio of *Ulva* spp.

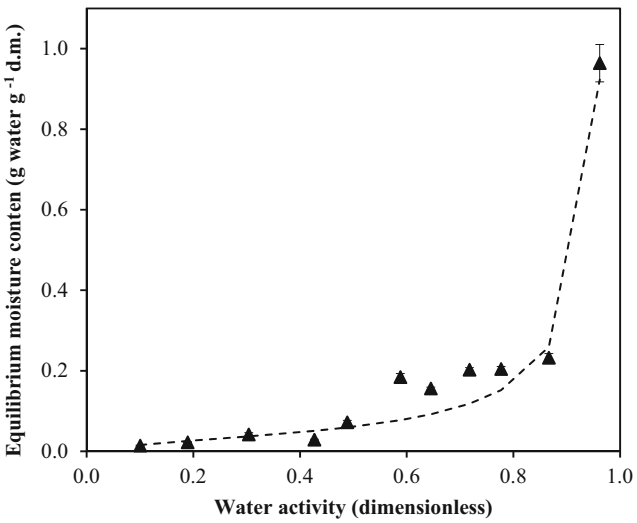

Fig. 1 Experimental data of equilibrium moisture contents for *Ulva* spp. desorption isotherm at 50 °C (▲). Lines (—) correspond to the BET model (Eq. 1). Values are averages ($n = 3$); error bars are standard deviation

Drying behavior of *Ulva* spp.

Figure 2 shows the curves of the moisture ratio versus the drying time (A), and the drying rate versus the

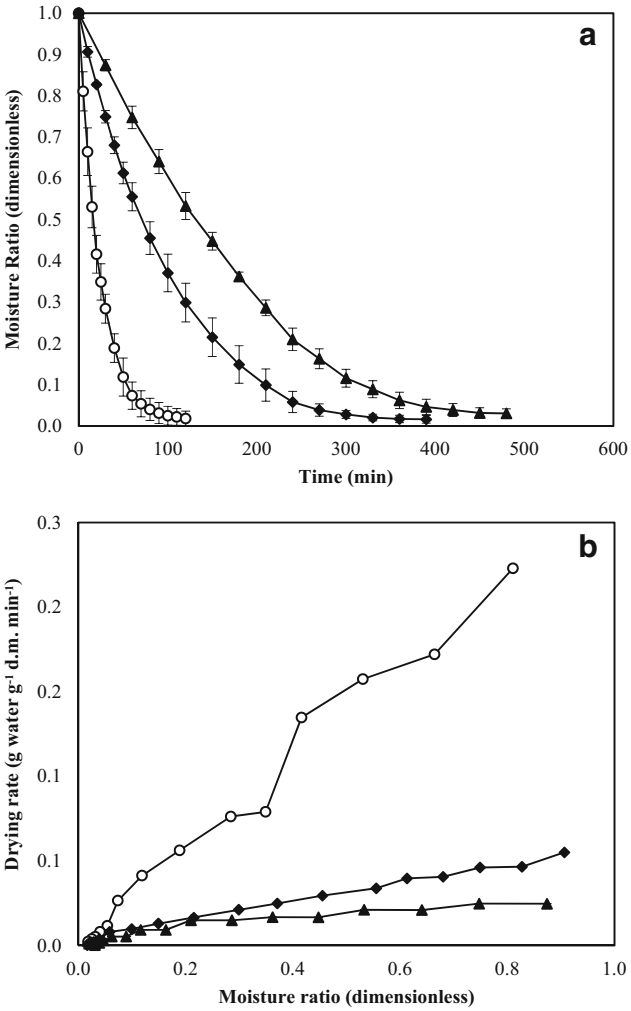

Fig. 2 **a** Drying behavior and **b** drying rates of *Ulva* spp. at different drying methods. Convective drying at 70 °C (O), vacuum drying at 70 °C (♦), and solar drying ~ 50 °C (▲). Values are averages ($n = 3$); error bars are standard deviation

moisture ratio (B) of dried *Ulva* spp. Moisture content is high during the initial phase of the drying process, which resulted in high drying rates due to higher moisture diffusion (Arslan and Özcan 2012). On the final phase of the drying process, the moisture ratio decreases to 0.018 in 120 min for CD, to 0.016 in 390 min for VD, and to 0.031 in 480 min for SD. As a result, drying time was dependent on the processing conditions employed, where fluctuating low temperature associated with SD resulted in longer drying time (Arslan and Özcan 2012). On the other hand, an increase of the drying temperature, like CD and VD, produced a reduction in drying time. Higher processing temperatures generate stronger driving forces for heat and mass transfer, which allows water molecules to escape easier and faster from the matrix of the product, speeding the drying process (Gupta et al. 2011).

It is noteworthy that CD shortened the drying time by 69.2 and 75.0% when compared to VD and SD, respectively (Fig. 2a). Therefore, *Ulva* spp. dried under convection had a significantly higher drying rate than the samples dried by vacuum or solar, being substantially higher in the first 30 min (Fig. 2b). This can be attributed to the fact that during CD, the continuous hot air circulation inside the oven removes faster the surface water from the vegetative thalli of *Ulva* due to a larger transfer area exposed to hot dry air. Results also indicate that VD gives a lower drying rate than CD. This can be associated to the inlet of cold air that is allowed in the drying chamber in the phase of atmospheric pressure, while the release valve remains open, whereas during the vacuum holding phase heated air is pumped out by vacuum pump, reducing the temperature of the drying chamber and biomass and thus decreasing the drying rate (Deng et al. 2017).

Effect of different drying methods on physicochemical composition of *Ulva* spp.

The proximate composition analysis and dietary fiber content of *Ulva* spp. subjected to different drying methodologies are presented in Table 2. For all dried samples of *Ulva* spp., moisture content and a_w were highly dependent on the drying methodology, varying from 6.00 to 0.97 and 0.309 to 0.050, respectively. All drying methodologies evaluated reduced a_w values below 0.6, as required for food products to control microbial growth and reduce enzymatic activity. The higher moisture content was for SD samples. A_w is a critical factor to ensure safety and quality of the seaweeds after harvest (Gupta et al. 2011) and plays an important role in determining their shelf life (Rohani-Ghadikolaei et al. 2012); thus, all drying methods could extend the shelf life of *Ulva* spp. since they reduced a_w in all samples, compared to fresh samples.

Fat content in dried *Ulva* spp. was slightly higher than in the one reported for the fresh sample (Table 1). No significant differences were found for FD, SD, and CD (average values of 3.25 g (100 g)$^{-1}$), while a slight increment (18%) was observed for VD. Some authors have reported changes in fat content during drying, associated to leakage or volatilization related to water loss (Wu and Mao 2008) which could be responsible for the observed changes. These results are in accordance with Hassan et al. (2007) who also reported difference in fat content of dried leaves under different methodologies. However, *Ulva* spp. could still be considered in the same range as cereals and legumes (< 2%), and only slightly higher than most land plants (0.2 to 1%) (Ortiz et al. 2009).

Ash content in dried *Ulva* spp. samples was affected by the drying methodology employed (Table 2). Ash content in SD and CD (average ash content of 19.65 g (100 g)$^{-1}$) was significantly higher than in FD and VD (9 and 14% reduction,

Table 2 Effect of different drying methods on proximate composition of dried *Ulva* spp.

Composition, g (100 g DM) $^{-1}$	FD	VD	SD	CD
Moisture[1]	0.97 ± 0.01^{d}	5.01 ± 0.09^{b}	6.00 ± 0.08^{a}	1.45 ± 0.01^{c}
Fat	3.29 ± 0.08^{b}	3.84 ± 0.20^{a}	3.11 ± 0.25^{b}	3.36 ± 0.19^{b}
Ash	18.09 ± 0.36^{b}	17.28 ± 0.15^{c}	19.81 ± 0.31^{a}	19.48 ± 0.55^{a}
Crude protein	15.90 ± 0.36^{c}	18.22 ± 0.01^{b}	16.48 ± 0.17^{c}	20.23 ± 0.82^{a}
Crude fiber	6.68 ± 0.27^{ab}	6.28 ± 0.19^{b}	6.98 ± 0.37^{a}	6.82 ± 0.18^{a}
Water activity, a_w[2]	0.050 ± 0.005^{d}	0.309 ± 0.003^{a}	0.284 ± 0.013^{b}	0.133 ± 0.015^{c}
Insoluble dietary fiber, IDF	27.81 ± 0.01^{a}	26.85 ± 1.55^{a}	25.41 ± 1.02^{a}	26.43 ± 0.41^{a}
Soluble dietary fiber, SDF	18.80 ± 1.36^{ab}	16.87 ± 0.91^{b}	17.73 ± 1.17^{ab}	20.73 ± 1.43^{a}
Total dietary fiber, TDF	46.61 ± 1.35^{a}	43.72 ± 2.47^{a}	43.14 ± 2.20^{a}	47.16 ± 1.02^{a}

Different letters in the same row indicate significant differences ($p < 0.05$) according to multiple range test (MRT). Standard deviation was calculated on three replicates. Values are expressed as mean ± standard deviation

FD freeze-drying, *VD* vacuum drying, *SD* solar drying, *CD* convective drying

[1] Expressed as g (100 g)$^{-1}$

[2] Dimensionless

respectively). Thus, after drying, ash is still one of the main components of *Ulva* spp., so the nutritional benefits associated to the mineral content were not strongly affected by the drying methodology.

Crude protein was elevated in dried samples of *Ulva*, with significant differences according to the drying methodology employed (Table 2). No significant differences were found for crude protein content for FD and SD (mean value of 16.19 g (100 g)$^{-1}$), while CD and VD were higher (25% and 12%, respectively). Changes in temperature and drying time are often associated to the degradation of proteins in food (Hassan et al. 2007), being the later more significant (Idah et al. 2010). In the present study, lower values of crude protein content were found in those methodologies with longer drying time, such as FD and SD.

Values of crude fiber content ranging from 6.28 to 6.98 g (100 g DM)$^{-1}$ were found for dried *Ulva* spp. in all drying methodologies studied (Table 2). Similar trend was observed for total dietary fiber (TDF), with no impact on the amount of TDF after drying (mean value: 45.16 g (100 g DM)$^{-1}$). On the other hand, insoluble dietary fiber (IDF) and soluble dietary fiber (SDF) were affected by the drying methodology employed. In all dried samples, IDF content was still higher than SDF, but the ratio IFD/SDF was reduced according to the processing conditions employed (1.48, 1.59, 1.43, and 1.27 for FD, VD, SD, and CD, respectively) when compared to fresh *Ulva* spp. These values are in agreement with Yaich et al. (2011) and Ortiz et al. (2006) who reported an IDF/SDF ratio of 1.67 and 1.22 for dried *U. lactuca*, respectively. Fiber content in seaweeds is composed of many soluble polysaccharides that are significantly different from the insoluble polysaccharides present in terrestrial plants (Ortiz et al. 2009). Therefore, part of the soluble fiber could be affected by the dehydration process during drying, as a result presenting differences in the fiber content of the samples; however, no reference of similar results were found in published articles. More research should be made in order to fully understand these results which are relevant since one of the main advantages of seaweeds is their fiber content.

Effect of different drying methods on color parameters of *Ulva* spp.

Color in foods is one of the most important parameters considered a decisive factor to establish quality, strongly related to appearance (Deng et al. 2017). Table 3 presents the color parameters for fresh and dried *Ulva* spp., which are consistent with bright green in all samples.

After drying, all the colorimetric coordinates were affected by the drying methodology used (Table 3). Brightness parameter (L^*) was significantly increased in FD samples when compared to the other dried samples. FD had the higher L* value, indicating that dried *Ulva* spp. has a brighter color. This is in agreement with Deng et al. (2017) who also reported an increment of L* after drying red pepper; however, results did not agree with regards to temperature increased since FD presented the higher L* and the lowest temperature. Tello-Ireland et al. (2011) also reported an increased in L^* after drying for the red seaweed *Gracilaria chilensis*. Both colorimetric parameters a^* and b^* showed significant differences among treatments. After drying, a^* parameter was increased, while b^* was reduced. Similar trend was found for dried *G. chilensis*, (Tello-Ireland et al. 2011). Those changes in the a* and b* parameters indicated that color in dried *Ulva* spp. samples was less yellow and more green, and are in agreement with the loss of bright green color due to the conversion of chlorophyll into pheophytin and pheophorbide, resulting in the formation of olive green pigments, or due to non-enzymatic browning (Tello-Ireland et al. 2011).

Color changes evaluation is more simple if parameters L*, a*, and b* are combined and presented as ΔE, C*, and h* (Table 3), according to Eq. (4), Eq. (5), and Eq. (6). Fresh samples of *Ulva* spp. presented C* value of 27.38 and h^* angle of − 1.20, which is consistent with dark green color.

Table 3 Effect of different drying methods on color parameters of *Ulva* spp.

Color parameter	Drying method				
	Fresh	FD	VD	SD	CD
L^*	27.77 ± 0.28	45.04 ± 1.01^{a}	31.20 ± 1.10^{c}	33.42 ± 0.80^{b}	35.20 ± 1.14^{b}
a^*	− 9.81 ± 0.18	−8.35 ± 0.10^{a}	− 6.11 ± 0.11^{d}	− 6.73 ± 0.13^{c}	− 7.29 ± 0.28^{b}
b^*	25.56 ± 1.11	18.34 ± 0.51^{a}	13.92 ± 0.61^{b}	14.92 ± 0.39^{b}	14.03 ± 0.86^{b}
ΔE	–	18.83 ± 0.85^{a}	12.75 ± 1.00^{c}	12.48 ± 1.04^{c}	14.03 ± 0.99^{b}
h^*	− 1.20 ± 0.02	− 1.14 ± 0.01^{a}	− 1.16 ± 0.01^{a}	− 1.15 ± 0.01^{a}	− 1.09 ± 0.01^{b}
C^*	27.38 ± 0.98	20.15 ± 0.51^{a}	15.21 ± 0.58^{b}	16.37 ± 0.40^{b}	15.81 ± 0.89^{b}

Different letters in the same row indicate significant differences ($p < 0.05$) according to multiple range test (MRT). Standard deviation was calculated on five replicates. Values are expressed as mean ± standard deviation

FD freeze-drying, *VD* vacuum drying, *SD* solar drying, *CD* convective drying

Freeze-dried *Ulva* spp. samples presented higher ΔE when compared to the other drying methodologies, followed by CD and VD and SD (with no differences among them). Similar results were found for C* and h* for FD *Ulva* spp. and the other dry samples.

Effect of different drying methods on bioactive compound content and antioxidant activity

Seaweeds are an important source of nutritive components and bioactive compounds with antioxidant activity. Seaweeds in their natural environment are constantly exposed to the oceanic conditions, which can include several hazards, including microorganisms, toxins, or adverse climatic conditions. These factors can lead to the formation of free radicals and other oxidizing compounds (Gupta et al. 2011). However, they show an amazing ability to adapt to those conditions due to their intrinsic chemical defense mechanism, mostly comprised of several secondary metabolites with antioxidant capacity (Peng et al. 2015).

TPC in dried *Ulva* spp. was significantly affected by the drying methodology (Table 4). SD had the highest TPC while CD presented the lowest content, with a 37% reduction of TPC compared to FD. Similar TPC were reported for dried seaweeds by other authors. Farasat et al. (2014) reported variable TPC for 5 different dried *Ulva* spp. ranging from 125.8 to 508 mg GAE g (100 g DM)$^{-1}$. On the other hand, Nwosu et al. (2011) reported very low quantity of TPC, compared to red and brown seaweeds. Gupta et al. (2011) reported higher values for TPC in brown seaweed (1550 mg GAE (100 g)$^{-1}$) and also reported a slight reduction of TPC after drying. The combination of high drying temperature and dehydration of the tissue could destroy part of the phenolic compounds. TFC was also measured in all dried samples and results are presented in Table 4. Farasat et al. (2014) reported TFC for different types of *Ulva*, ranging from 804.8 to 3309.4 mg rutin equivalent per 100 g DM. Gupta et al. (2011) reported 490 mg quercitin equivalents per 100 g DM for fresh brown seaweed. Drying affected TFC in all samples, where VD and CD presented the highest TFC followed by FD and SD, with a reduction of 25 and 33%, respectively.

Seaweed can be classified according to three major divisions based in main color: Rhodophyta (red seaweeds), Chlorophyta (green seaweeds), and Chromophyta (brown, golden, yellow-green seaweeds. While all of them contain chlorophyll *a*, only green seaweeds also contain chlorophyll *b* (Hamid et al. 2015). Samples of *Ulva* spp. contain both types of chlorophyll (Table 4). Chakraborty and Santra (2008) reported values of 2060 and 1350 μg g^{-1} DM for Chl*a* and Chl*b* in *U. lactuca*. Rohani-Ghadikolaei et al. (2012) also reported values of Chl*a* markedly higher (3500 μg g^{-1} DM for *U. lactuca*). These discrepancies could be attributed to geographical location and species variability, but more importantly, to a difference on the extraction of pigments from the tissue. Both Chl*a* and Chl*b* were higher in FD *Ulva* spp. than in the other dried samples, while SD had the lowest Chl*a* content and VD, the lowest Chl*b* content.

Carotenoids, in addition to the contribution to the attractive color of vegetables, stand out due to their physiological effect as they are commonly associated with several health benefits, such as cancer prevention and pro-vitamin A activity (Vimala et al. 2011). It is important that carotenoids are included in the diet of humans and animals because they cannot synthesize them, as a result food fortification, dietary diversification. In seaweeds, carotenoids take part in photosynthesis and also act as antioxidant compounds that inactivate free radicals. Samples of dried *Ulva* spp. had a TCC ranging from 470.1 to 887.8 μg (g DM)$^{-1}$, with significant differences among drying methods (Table 4). These values were significantly higher than previously reported data. Ortiz et al. (2009) reported 198.6 μg (g DM)$^{-1}$ for a green seaweed (*C. fragile*) and Chakraborty and Santra (2008) reported

Table 4 Effect of different drying methods on total phenolic content (TPC), total flavonoid content (TFC), photosynthetic pigment concentrations, and antioxidant capacity of dried *Ulva* spp.

Parameters	FD	VD	SD	CD
TPC, mg GAE (100 g DM)$^{-1}$	134.56 ± 5.47^{b}	129.08 ± 7.34^{b}	143.79 ± 5.76^{a}	90.24 ± 4.84^{c}
TFC, mg CE (100 g DM)$^{-1}$	22.91 ± 2.23^{b}	31.32 ± 1.98^{a}	20.62 ± 0.77^{c}	30.01 ± 2.25^{a}
Chlorophyll *a*, μg g^{-1} DM)	1582.1 ± 14.6^{a}	1036.7 ± 16.7^{b}	955.1 ± 68.9^{c}	1022.1 ± 35.7^{bc}
Chlorophyll *b*, μg g^{-1} DM	1923.6 ± 55.7^{a}	347.5 ± 1.4^{c}	522.2 ± 3.6^{b}	488.0 ± 12.5^{b}
Total carotenoids, μg g^{-1} DM	887.8 ± 78.3^{a}	523.5 ± 11.4^{bc}	470.1 ± 25.0^{c}	583.8 ± 12.9^{b}
DPPH, μmol TE (100 g DM)$^{-1}$	282.3 ± 10.4^{a}	187.7 ± 10.5^{c}	238.6 ± 13.2^{b}	223.1 ± 18.7^{b}
ORAC, μmol TE (100 g DM)$^{-1}$	3245.4 ± 200.4^{b}	3980.7 ± 357.3^{a}	3484.9 ± 156.4^{ab}	4080.9 ± 321.9^{a}

Different letters in the same row indicate significant differences ($p < 0.05$) according to multiple range test (MRT). Standard deviation was calculated on three replicates. Values are expressed as mean ± standard deviation

TPC total phenolic content, *TFC* total flavonoid content, *DPPH* 2,2-diphenyl-1-picrylhydrazyl, *ORAC* radical absorbance capacity, *GAE* gallic acid equivalents, *CE* catechin equivalents, *TE* trolox equivalents, *FD* freeze-drying, *VD* vacuum drying, *SD* solar drying, *CD* convective drying

19.55 μg (g DM)$^{-1}$ for *U. lactuca*. The degradation of carotenoids probably occurred due to oxidation (Vimala et al. 2011). FD gave the highest TCC, followed by VD and CD (38% reduction) and SD (47% reduction). Lower and more controlled temperature of the drying process may be responsible for the retention of TCC in FD.

The antioxidant activity of dried samples of *Ulva* spp. by DPPH and ORAC methodologies are reported in Table 4. The antioxidant capacity of dried *Ulva* spp. measured by the DPPH assay decreased when compared to fresh samples (Table 1), as reported by Tello-Ireland et al. (2011). Vega-Gálvez et al. (2008) also found that antioxidant capacity was reduced in red pepper after drying. Deng et al. (2017) stated that reduction of antioxidant capacity on dried samples of red pepper is associated to high temperature processing and to the duration of the drying cycle. Different trends were obtained for antioxidant capacity by DPPH and ORAC assays for dried *Ulva* spp.; both reduced the antioxidant capacity compared to fresh samples, where DPPH showed a mean reduction close to 80% whereas ORAC reduction was only 10%. FD resulted in a higher antioxidant capacity by DPPH, while CD resulted in the highest ORAC antioxidant capacity of dried *Ulva* spp. The differences in the behavior of the antioxidant capacity could be explained because each assay is based on a different chemical system and/or reaction. For instance, DPPH assay is based on electron transfer (ET). This assay estimates the capacity of an antioxidant to reduce an oxidant that changes in color when reduced. Conversely, the ORAC assay is based on hydrogen atom transfer (HAT) reactions, in which antioxidant and substrate compete for thermally generated peroxyl radicals through the decomposition of azo compounds (Prior 2015).

Due to their nutritional and pharmaceutical value, seaweeds can be consumed as food or used as medicine, condiments, or dietary supplements, among others (Peña-Rodríguez et al. 2011). Even if drying had an effect on the bioactive compounds content and their antioxidant capacity, dried *Ulva* spp. was able to retain most of the nutritional and functional characteristics and could be employed as an interesting functional ingredient.

Effect of different drying methods on fatty acid composition of *Ulva* spp.

Even if green seaweeds have a low fat content, they are important functional ingredients because they have higher content in saturated and unsaturated fatty acids than most common terrestrial plants (Hamid et al. 2015). FA composition of dried *Ulva* spp. is presented in Table 5. Eighteen different FAs

Table 5 Effect of different drying methods on fatty acid composition of *Ulva* spp.

Fatty acid, g (100 g of total fatty acid)$^{-1}$	Drying methods			
	FD	VD	SD	CD
Caprylic acid C8:0	2.93 ± 0.02^{c}	4.73 ± 0.34^{b}	6.53 ± 0.41^{a}	4.65 ± 0.33^{b}
Lauric acid C12:0	0.75 ± 0.12^{b}	0.88 ± 0.03^{b}	1.12 ± 0.03^{a}	0.86 ± 0.10^{b}
Myristic acid C14:0	0.18 ± 0.00^{c}	0.25 ± 0.02^{b}	0.32 ± 0.01^{a}	0.24 ± 0.00^{b}
Myristoleic acid C14:1	0.59 ± 0.01^{ab}	0.56 ± 0.02^{b}	0.63 ± 0.06^{a}	0.58 ± 0.02^{ab}
Palmitic acid C16:0	19.49 ± 2.08^{a}	18.47 ± 1.81^{a}	21.33 ± 2.43^{a}	17.55 ± 2.06^{a}
Palmitoleic acid C16:1	0.87 ± 0.08^{a}	0.88 ± 0.09^{a}	0.84 ± 0.05^{a}	0.93 ± 0.04^{a}
Stearic acid C18:0	0.64 ± 0.09^{a}	0.47 ± 0.07^{b}	0.73 ± 0.12^{a}	0.31 ± 0.01^{c}
Oleic acid C18:1ω9	1.57 ± 0.04^{a}	1.28 ± 0.07^{bc}	1.17 ± 0.14^{c}	1.38 ± 0.06^{b}
Oleic acid C18:1ω11	7.87 ± 0.63^{a}	7.83 ± 0.62^{a}	7.78 ± 0.91^{a}	7.57 ± 0.60^{a}
Linoleic acid C18:2ω6	17.12 ± 0.58^{ab}	18.29 ± 0.41^{a}	16.46 ± 1.72^{b}	18.75 ± 0.57^{a}
Gamma-linolenic acid C18:3ω6	0.91 ± 0.04^{a}	0.83 ± 0.03^{bc}	0.77 ± 0.06^{c}	0.84 ± 0.01^{ab}
Alpha-linolenic acid C18:3ω3	22.15 ± 0.70^{a}	21.80 ± 0.84^{ab}	19.90 ± 1.04^{b}	21.27 ± 1.58^{ab}
Docosanoic acid C22:0	0.84 ± 0.05^{b}	0.85 ± 0.05^{b}	1.16 ± 0.08^{a}	0.83 ± 0.02^{b}
Dihomo-gamma-linolenic acid C20:3ω6	0.22 ± 0.02^{c}	0.32 ± 0.00^{ab}	0.34 ± 0.00^{a}	0.29 ± 0.03^{b}
Eicosatrienoic acid C20:3ω3	0.21 ± 0.01^{b}	0.16 ± 0.00^{d}	0.28 ± 0.01^{a}	0.19 ± 0.01^{c}
Arachidonic acid C20:4ω6	0.83 ± 0.08^{b}	0.96 ± 0.05^{a}	0.97 ± 0.02^{a}	0.90 ± 0.07^{ab}
Eicosapentaenoic acid C20:5ω3	0.51 ± 0.01^{c}	0.63 ± 0.03^{b}	0.60 ± 0.06^{b}	0.70 ± 0.00^{a}
Nervonic acid C24:1	2.72 ± 0.14^{b}	2.55 ± 0.13^{b}	3.07 ± 0.06^{a}	2.67 ± 0.09^{b}
SFA[1]	24.82 ± 2.08^{b}	25.65 ± 1.47^{b}	24.44 ± 1.82^{b}	
MUFA[2]	13.63 ± 0.58^{a}	13.10 ± 0.68^{a}	13.49 ± 0.91^{a}	13.13 ± 0.59^{a}
PUFA[3]	41.94 ± 0.25^{a}	43.00 ± 0.57^{a}	39.32 ± 0.75^{b}	42.94 ± 2.25^{a}
ω3/ω6	1.20 ± 0.07^{a}	1.07 ± 0.03^{a}	1.13 ± 0.18^{a}	1.07 ± 0.04^{a}

Different letters in the same row indicate significant differences ($p < 0.05$) according to multiple range test (MRT). Standard deviation was calculated on three replicates. Values are expressed as mean ± standard deviation

FD freeze-drying, *VD* vacuum drying, *SD* solar drying, *CD* convective drying

[1] Saturated fatty acids: C8:0, C12:0, C14:0, C16:0, C18:0, and C22:0

[2] Monounsaturated fatty acids: C14:1, C16:1, C18:1ω9, C18:1ω11, and C24:1

[3] Polyunsaturated fatty acids: C18:2ω6, C18:3ω6, C18:3ω3, C20:3ω6, C20:3ω3, C20:4ω6, and C20:5ω3

Table 6 Effect of drying methods on amino acid profile of *Ulva* spp

Amino acids, g (100 g protein)$^{-1}$	FD	VD	SD	CD
Aspartic acid	3.72 ± 0.06^{a}	1.94 ± 0.18^{c}	1.87 ± 0.18^{c}	2.58 ± 0.15^{b}
Glutamic acid	2.04 ± 0.11^{b}	3.96 ± 0.46^{a}	4.08 ± 0.23^{a}	1.92 ± 0.14^{b}
Serine	5.78 ± 0.05^{a}	5.18 ± 0.36^{b}	5.17 ± 0.07^{b}	3.79 ± 0.22^{c}
Glycine	20.83 ± 0.20^{a}	17.31 ± 1.22^{b}	14.89 ± 0.52^{c}	13.39 ± 0.46^{d}
Histidine	ND^{b}	4.11 ± 0.13^{a}	ND^{b}	ND^{b}
Arginine	11.20 ± 0.31^{a}	9.55 ± 0.42^{b}	9.29 ± 0.48^{b}	7.16 ± 0.24^{c}
Threonine	8.51 ± 0.25^{a}	7.33 ± 0.32^{b}	7.13 ± 0.36^{b}	5.51 ± 0.17^{c}
Alanine	10.48 ± 0.38^{a}	4.91 ± 0.10^{c}	4.33 ± 0.34^{d}	7.27 ± 0.03^{b}
Proline	13.74 ± 0.45^{b}	19.78 ± 0.24^{a}	15.24 ± 1.79^{b}	9.59 ± 0.03^{c}
Tyrosine	39.20 ± 1.48^{a}	21.61 ± 1.45^{b}	19.11 ± 1.42^{b}	21.54 ± 1.30^{b}
Valine	4.17 ± 0.27^{a}	3.48 ± 0.05^{b}	2.70 ± 0.16^{c}	2.94 ± 0.26^{c}
Methionine	8.12 ± 0.45^{a}	7.78 ± 0.41^{a}	5.76 ± 0.39^{b}	6.29 ± 1.12^{b}
Cysteine	8.17 ± 0.70^{a}	4.55 ± 0.59^{bc}	4.06 ± 0.31^{c}	5.33 ± 0.12^{b}
Isoleucine	3.46 ± 0.30^{a}	3.21 ± 0.11^{a}	3.50 ± 0.26^{a}	2.28 ± 0.03^{b}
Leucine	7.22 ± 0.91^{a}	7.38 ± 0.81^{a}	7.40 ± 0.30^{a}	5.33 ± 0.12^{b}
Phenylalanine	14.28 ± 1.48^{b}	17.22 ± 0.12^{a}	13.72 ± 0.13^{bc}	12.16 ± 1.12^{c}
Lysine	6.49 ± 0.88^{b}	6.21 ± 0.10^{b}	7.91 ± 0.55^{a}	6.86 ± 0.29^{b}
Total EAA[1]	52.23 ± 4.55^{a}	56.71 ± 1.91^{ab}	48.13 ± 2.15^{a}	41.38 ± 3.50^{c}
Total AA[2]	167.39 ± 8.27^{a}	145.50 ± 7.05^{b}	126.15 ± 7.48^{c}	113.96 ± 6.20^{c}

Different letters in the same row indicate significant differences ($p < 0.05$) according to multiple range test (MRT). Standard deviation was calculated on three replicates. Values are expressed as mean ± standard deviation

FD freeze-drying, *VD* vacuum drying, *SD* solar drying, *CD* convective drying, *Nd* not determined

[1] Total essential amino acids: Thr, Val, Met, Ile, Leu, and Lys (excluding Trp and His)

[2] Total amino acids (excluding Trp and His)

were identified in the dried samples of *Ulva* spp., six of them are saturated fatty acids (SFA), five are monounsaturated fatty acids (MUFA), and seven are polyunsaturated fatty acids (PUFA). Alpha-linolenic acid, palmitic acid, and linoleic acid are the main fatty acids in dried *Ulva* spp., regardless of the drying methods employed. Yaich et al. (2011) reported that palmitic acid (59.40%) and oleic acid (15.93%) were the most abundant in the case of *U. lactuca*. In all dried *Ulva* spp. samples, several essential fatty acids were present, such as linoleic acid, linolenic acid, and eicosanoid precursors (arachidonic acid, eicosapentaenoic acid). As a result, *Ulva* spp. could have applications in the pharmaceutical and food industries due to their content of C18 PUFAs, which are considered essential to humans (Yaich et al. 2011).

Low consumption of saturated fat (from animal sources) and increased consumption of foods with a high PUFA/SFA ratio are associated with a lower risk of coronary heart diseases; thus, the PUFA/SFA ratio is one of the parameters used to assess the nutritional quality of the lipid fraction of foods (Chan and Matanjun 2017). In all dried samples, PUFA represented the main portion (52, 53, 47, and 53% for FD, VD, SD, and CD, respectively) (Table 5). The second main portion was comprised of SFA with a 31, 31, 37, and 30% for FD, VD, SD, and CD, respectively. The dominance of unsaturated fatty acids over saturated fatty acids, and the prevalence of PUFA over MUFA among the unsaturated is in accordance with previous studies (Ortiz et al. 2006). These results are similar to those reported by Chan and Matanjun (2017) for the red seaweed *Gracilaria changii* (50% of PUFAs).

Seaweeds are known for their lipid quality, especially for the *ω3/ω6* ratio. As shown in Table 3, *Ulva* spp. contain several *ω3*-PUFAs, known to prevent the growth of atherosclerotic plaque in blood vessels, reducing blood pressure, and improving the immune function, and *ω6*-PUFAs that are responsible to maintain a healthy ratio between high- and low-density cholesterol. Therefore, it is important to maintain a balanced consumption of ω-6 and ω-3 PUFAs in diet based on a ratio of $\omega 6/\omega 3 < 10$ recommended by the WHO (Chan and Matanjun 2017). Moreover, Hamid et al. (2015) claimed that the prehistoric diet of humans had a ω6/ω3 ratio of approximately 1:1, closer to the current Mediterranean diet (which is considered healthy). In the present study, ω3/ω6 ratio was not affected by the drying processes employed, obtaining a ω3/ω6 ratio close to one for all treatments. Similar ω3/ω6 ratio was previously reported for green seaweed *U. lactuca* by Ortiz et al. (2006). The fatty acid profile of dried *Ulva* spp. indicates its potential as a functional food, due to a high amount of essential PUFAs and a favorable ω3/ω6 ratio.

Effect of different drying methods on the amino acid composition of *Ulva* spp.

The amino acid composition of all dried seaweeds under different drying conditions is reported in Table 6. The results in this study showed that dried *Ulva* spp. (regardless of the drying method used) contained glycine, proline, tyrosine, and phenylalanine and lower amounts of aspartic acid, glutamic acid, and isoleucine. This is different from previous studies that have reported high levels of aspartic and glutamic acids in *U. lactuca* (Ortiz et al. 2006; Yaich et al. 2011; Shuuluka et al. 2013), *U. clathrata* (Peña-Rodríguez et al. 2011), and *U. capensis* and *U. rigida* (Shuuluka et al. 2013). It has been well known that amino acids are susceptible to drying technologies and could be lost, changed, or even destroyed during processing. Indeed, in the current study, histidine was determined only in the VD sample. Histidine is a very oxidation sensitive amino acid; however, moisture removal in the VD process occurs in the absence of oxygen, resulting in minimized oxidative degradations (Piwinska et al. 2015). The highest degradation ratio of amino acid with respect to FD sample was found in CD product (31.9%) followed by SD (24.6%) and VD (13.1%). This indicates that heat treatment may change the composition of nitrogenous compounds. A similar trend was also reported by Chan et al. (1997) and Wong and Cheung (2001) for *Sargassum* species.

Conclusion

The proximate composition analyses of fresh *Ulva* Spp. showed that ash, protein, and crude fiber are the main constituents, without considering water content. A typical type II isotherm was found and the BET model gave the better fit for the experimental data. Drying methodology had an effect on the proximate composition of *Ulva* spp., being convective drying the method that better maintained the physicochemical and functional parameters. The samples of dried *Ulva* spp. presented a high amount of total dietary fiber (with an IFD/SFD ~ 1.5), fatty acids (> 50% of them unsaturated, with a ω3/ω6 ~ 1:1) and a high proportion of essential amino acids. Other minor constituents identified in all dried *Ulva* spp. samples with functional relevance were phenolic compounds, flavonoids, and carotenoids. The fatty acid and amino acid profiles showed the presence of several essential polyunsaturated fatty acids and amino acids of nutritional importance.

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Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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