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# Comparative evaluation of physicochemical profile and bioactive properties of red edible seaweed *Chondrus crispus* subjected to different drying methods

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ABSTRACT

Dehydration of the edible seaweed *Chondrus crispus* was performed by freeze-drying, conventional oven-drying and emerging microwave hydrodiffusion and gravity (MHG). In this work, the drying kinetics and modelling, estimating specific energy consumption and environmental impact of distinct processes were tested. Color and microstructural features of the dried macroalgae were also evaluated, as well as their nutritive characterization, chemical profile and bioactive potential (antioxidant and antimicrobial activities). Moreover, collected liquid phases from both the defrosted and MHG treated samples were also characterized. All methodologies provided solid phases with an adequate final moisture content. MHG significantly reduced the needed time, specific energy consumption and environmental impact, providing *C. crispus* with intermediate color and histological structure characteristics. Overall, this trend was also defined to tested chemical parameters and bioactivities. MHG provided aqueous extracts with potential bioactive compounds from this red alga, increasing the efficiency of this drying method.

## 1. Introduction

Harvesting and human consumption of edible macroalgae resources have primarily been associated with the population of marine regions for thousands of years, since ancient times (Wells et al., 2017). Currently, their nutritional value is globally recognized favoring their widespread use, including European, South American countries and United States of America. Nowadays, scientific evidence confirmed that their regular intake can provide a large variety of potential functional benefits on human health, such as prevention of cardiovascular dysfunctions, diabetes mellitus and obesity-related diseases. The algal biological properties are based in their bioactive compounds with anti-inflammatory, antimicrobial, antioxidant or antitumor characteristics, among other profits. These benefic biological effects confer to seaweed a relevant role in health-related food habits which are extended to cosmetic, medical, pharmacological and allied sectors due to their therapeutic features (Ganesan et al., 2019).

The high moisture content and nutrient components of the sea vegetables are the main factors that causes their damage by microbial growth and restricts the shelf life of these perishable marine bioresources. The establishment of preservation methodologies could ensure their access independently of the season period and geographical limitations. In this context, some stabilization techniques have been utilized in combination with refrigerated and freezing storage as for example salting, dehydration methodologies or high-pressure processing (del Olmo et al., 2020). The reduction of algae water activity by drying procedures is widely used in order to remove the portion of the present free water and, in consequence, secure the food safety of these foodstuffs. In addition, their storage cost is reduced because this can be development at room temperature and the experimented algae

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reduction of bulk and weight facilitates their transport. Hot air drying process is the drying methodology more prevalent in food industry; other convective drying preservation techniques are also freeze-drying, heat pump drying, spray-drying and superheated steam drying procedures. Furthermore, conductive and radiative drying methods are possible procedures to treat algae biomass. Specifically, infrared, microwave, solar and ultrasound radiations can improve the drying conditions (Bennamoun & Li, 2018). The employment of emerging technologies enhances drying efficiency rates at the same time, the sustainability of the process is supported since the drying process can be carried out with lower inputs. Regarding the study of the application of microwave irradiation technology to macroalgae drying is restricted since their evaluation was only tested to *Gracilaria* (Jin et al., 2015), *Laminaria* (López-Hortas et al., 2018) and *Undaria* (López-Hortas et al., 2019b) species using microwave vacuum drying and microwave hydrodiffusion and gravity (MHG) technologies, respectively. This last innovative method was applied in the current work to *Chondrus crispus* (Gigartinales, Rhodophyta) for first time in red seaweed samples according to the information to which the authors have access. The potential of this novel technology on the treatment of *C. crispus* as representative of other carrageenophyte seaweeds was compared with samples processed by freeze-drying and oven-drying conventional procedures in order to determine the most attractive procedure to optimize the optimum food preservation conditions. Moreover, drying kinetics and their corresponding modelling as well as the estimated specific energy consumption and environmental impact of the different methods were provided in addition to their color features and microstructural changes. The impact of these three drying methods on the nutritive value of this red macroalga was tested by an in-depth chemical characterization and evaluation of its *in vivo* antioxidant bioactivity and antimicrobial properties. Furthermore, the recovered extracts by defrosting phase and MHG were characterized to define their antioxidant capacity and physicochemical profile.

## 2. Materials and methods

### 2.1. Raw materials and reagents

An overview of the executed research by the drying of *C. crispus* seaweed is shown in the flow diagram of Fig. 1. Concerning that, this scheme also gathers the characterization of the collected solid and liquid phases from this procedure. Wild *C. chondrus* red seaweed samples (moisture content 74.72 ± 0.05% wet basis (w.b.)) were purchased in November 2019 by *Conservas Mar de Ardora Company* (A Coruña, Spain). These samples were gathering on the low-shore zone of Galician coastline submitted to Atlantic influence (Northwest Spain). Fresh seaweeds (5 kg) were identified, collected, packed into plastic bags properly marked and kept at 5 °C into an expanded polystyrene thermic box for

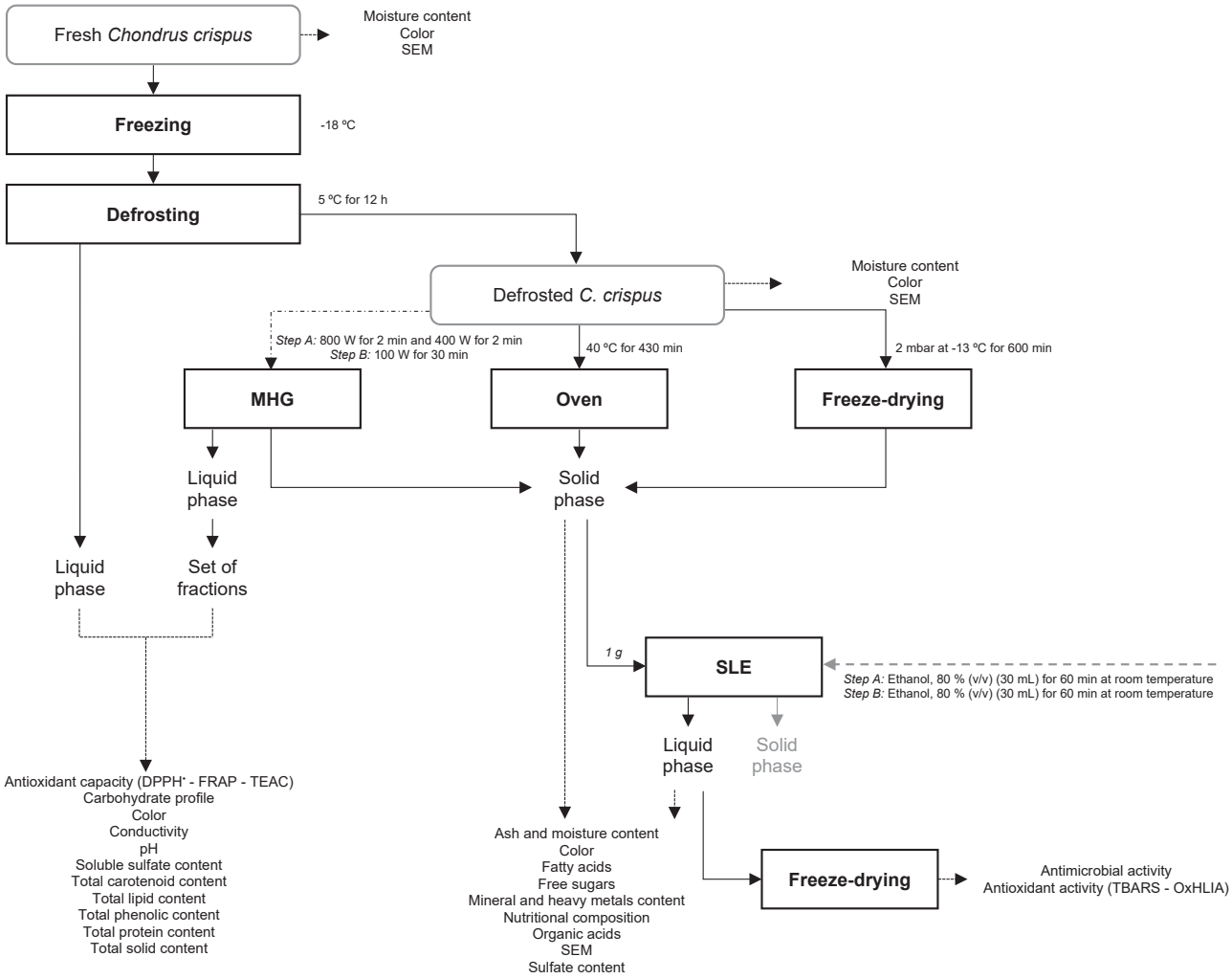


**Fig. 1.** Flowchart of studied dehydration processes of edible *Chondrus crispus* red seaweed. DPPH•: 1,1-Diphenyl-2-picryl-hydrazyl; FRAP: Ferric reducing antioxidant power; MHG: Microwave hydrodiffusion and gravity; OxHLIA: Oxidative haemolysis inhibition; SEM: Scanning electron microscopy; SLE: Solid-liquid extraction; TBARS: Thiobarbituric acid reactive substances; TEAC: Trolox equivalent antioxidant capacity.

their transport to their reception in the laboratory, which was made within 24 h. Samples were carefully selected, packed in closed plastic bags, protected from light and stored at −18 °C until further analysis, within a period of two months from the date of gathering. Before drying the algae, it was necessary submitted the samples to a defrosted step at 5 °C for 12 h reserving the seaweed samples (moisture content 71.88 ± 0.97% w.b.) and the generated defrosting liquid phase. All utilized chemicals reagents were of analytical grade or the highest available grade.

### 2.2. Seaweed processing

Defrosted wild-harvested *C. crispus* seaweed was placed in toroidal geometry (load density of 664.81 ± 0.01 kg/m$^3$) in a NEOS-GR MA126 equipment (Milestone Srl, Italy) employing a discontinuous optimized combination of irradiation power by MHG technology (first step: 800 W for 2 min and below 400 W for 2 min; second step: 100 W for 30 min). Samples were taken off the system for 30 min between these two phases in order to prevent that the generated residual heat burned them. Pre-dried seaweed was also manually rotated on themselves to homogenize their drying treatment in this intermediate step. These working conditions were based on several previous laboratory experiments as well as recent findings (López-Hortas et al., 2018). Aqueous extracts, collected during the treatment in fractions of 1 mL by gravity, were mixed and stored at 4 °C in the light absence until their analysis. Dried MHG samples were compared with the solid phases obtained by freeze-drying and oven-drying methods. Sublimation and vacuum pump of freeze-drying was conducted on a vacuum lyophilizer Alpha 2–4 LDplus (Martin Christ Gefriertrocknungsanlagen GmbH, Germany) at a reduced pressure of 2 mbar and −13 °C (load density of 316.90 ± 0.01 kg/m$^3$) until constant weight was achieved. On the other hand, samples were placed on a tray (2277.36 ± 0.01 cm$^2$) forming a monolayer (load density of 0.99 ± 0.01 kg/m$^2$) and treated in a laboratory drying oven (JP Selecta, S.A., Spain) at 40 ± 2 °C. Oven-dried samples were periodically weighed until weight became constant. Dried samples were placed into a desiccator containing silica gel for 12 h in order to cool and homogenize their moisture content. All experiments were at least performed in triplicate.

### 2.3. Yield of drying tested procedures

The recorded variation of samples mass before and after drying was used to evaluate their yield in percentage following reported work (Mondal et al., 2019), eq. (1),

$$\text{Yield}\ (\%) = \frac{M_s}{M_f} \times 100 \quad (1)$$

where $M_s$ e $M_f$ are the mass of dried and defrosted seaweed samples, respectively.

### 2.4. Drying kinetics and mathematical modelling

The relative water removal or moisture ratios (MR) from dried *C. crispus* by tested drying methods was defined using eq. (2),

$$MR = \frac{M_t - M_e}{M_0 - M_e} \quad (2)$$

being $M_t$ the moisture content (kg water/(kg dry basis, d.b.)) of the samples at any drying time, $M_0$ the initial moisture content and $M_e$ the equilibrium moisture content.

Page model of two-parameters was employed for the modelling of experimental drying kinetics (Page, 1949), namely eq. (3),

$$MR = e^{-kt^n} \quad (3)$$

where k (min$^{-n}$) and n (-) display the corresponding model parameters, whereas t (min) means dehydration time.

### 2.5. Estimated operational cost and environmental impact

An estimation of specific electrical energy consumption, as the main critical factor of the operating costs, was carried out following the next equation, eq. (4),

$$E = tP \quad (4)$$

being *E* the specific energy consumption (J), *t* the processing time (s) and *P* the utilized irradiation power (W). For this estimation, only drying processing steps were considered so the generated consumption of previous freezing, necessary to freeze-dried technology, was not measured.

The determination of the operating cost of this energy requirements was calculated considering the unit price of electrical energy for non-household users (0.1254 €/kWh) of in European Union market in first half of 2020 (Eurostat, 2021).

The consumption of 1 kWh from coal or fuel was associated with the released of 800 g of dioxide carbon so the environmental impact of the different treatments can be considered (Benmoussa et al., 2018).

### 2.6. Dried C. crispus seaweed characterization

#### 2.6.1. Physical features

International Commission on Illumination (CIE, Commission internationale de l'éclairage) color space $L^*a^*b^*$ were recorded using a portable colorimeter CR-400 (Konica Minolta, Japan) with D65 as standard illuminant equipped with a pulsed xenon lamp to obtain color coordinates (lightness, $L^*$, (degree of whiteness, $0 < L^* < 100$, brightness), $a^*$ (redness ($a^* > 0$) or greenness ($a^* < 0$) degree) and $b^*$ (yellowness ($b^* > 0$) or blueness ($b^* < 0$) degree), respectively). The color magnitudes (hue-angle (h* (°)), Chroma (C*) and saturation (S*)) as well as total color and hue differences (ΔE* and ΔH*, in that order) were calculated in accordance with the following equations, eq. (5–9),

$$h^*(°) = \arctan\left(\frac{b^*}{a^*}\right) \quad (5)$$

$$C^* = \sqrt{\left(a^{*2} + b^{*2}\right)} \quad (6)$$

$$S^* = \frac{C^*}{L^*} \quad (7)$$

$$\Delta E^* = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \quad (8)$$

$$\Delta H^* = \sqrt{(\Delta E^*)^2 - (\Delta L^*)^2 - (\Delta C^*)^2} \quad (9)$$

Note here that these same indications were also utilized to describe the color features of the recovered liquid samples.

The comparison of the surface tissue structure of the seaweed untreated, defrosted and dried samples was performed by scanning electron microscopic (SEM) observation. For this purpose, representative portions of algae were suitably freeze-dried to preserve the absence of distortion in these tissues as effect of water removal process caused by the high vacuum required in their treatment. Samples were inserted onto aluminum stubs and submitted to a metal coating with gold using an Emitech K550X (Quorum Technologies Ltd, UK) equipment. The observations were carried out at least in duplicate by Field Emission Ion (FEI) QUANTA 200 (FEI Company, The Netherlands) electron scanning microscope. The magnification of the images was in a range from 1000 to 6000 times and these were taken under vacuum conditions at an accelerating voltage of 15.0 kV.

*2.6.2. Nutritional profile and chemical properties*

Main nutritional values were defined by the chemical characterization of the different dried samples. Moisture and ash content were determined gravimetrically by standard protocols by the Association of Official Analytical Chemists (2019) at 105 °C for 24 h and 575 °C for 6 h, respectively. Following these analytical methods, the fat content of tested samples was analyzed by Soxhlet extraction with petroleum ether as solvent and the protein content of dried samples was examined by Kjeldahl method, using the corresponding red seaweeds conversion factor of 4.59 following instructions from Lourenço et al. (2002). Their total carbohydrate content was taken out by the difference with other components. Additionally, the seaweeds energetic value was estimated according to the conversion method using Atwater's factors, reported elsewhere (Fernandes et al., 2021).

The characterization of the elemental carbon fraction was measured by flash combustion technology using a gas chromatographic analyzer with a thermal conductivity detector. The presence of C corresponded with the identified $CO_2$ fraction (Barral-Martínez et al., 2020). High-performance liquid chromatography (HPLC) instrument equipped with Smartline system 1000 pump, Smartline Manager 5000 degasser, Jasco AS-2057 autosampler and Smartline 2300 refractive index detector (Knauer (Germany) and Jasco, Easton (USA)) was employed to analyze free sugar profile following working conditions described by by Fernandes et al. (2021). The analytical methodologies to define the organic acids content and fatty acids methyl esters profile indicated in this report was also utilized by characterize the concentration of these compounds in *C. crispus* seaweed. For this reason, ultra-fast liquid chromatography system Shimadzu 20A series integrated by DGU-20A degasser, Nexera SIL-20A autosampler with temperature-controlled tray, CTO-20AS column oven and SPD-M20A photodiode array detector (Shimadzu Corporation, Japan) were carried out to analyze organic acids profile of tested samples. Furthermore, gas chromatography DANI model GC 1000 instrument with a flame ionization detector (Contone, Switzerland) was employed to define the fatty acids content of red macroalgae samples. Summarily, the lipid fraction sample, obtained from previous Soxhlet extraction using petroleum ether, was mixed with methanol:sulphuric acid 95%:toluene (2:1:1, v/v/v) solvent for 12 h at 50 °C and 160 rpm. Distilled water and diethyl ether were added in order to facilitate the later separation of the target upper phase for their posterior analysis. Ionic chromatography test was utilized to determine the seaweed sulfate content following as previously detailed by Barral-Martínez et al. (2020). Note here that the retention time of the different compounds was employing to the identification of the different target compounds and their quantification was performed employing internal standard method as well as specific calibration curves.

Mineral (calcium, chromium, copper, iron, magnesium, phosphorus, potassium, sodium, and zinc) and heavy metals (arsenic, cadmium, iodine, lead and mercury) content of the samples were examined after their acidic digestion process by microwave treatment as described by Barral-Martínez et al. (2020). For this purpose, inductively coupled plasma-atomic emission spectrophotometry, inductively coupled plasma-mass spectrophotometry and cold vapor-atomic absorption spectrophotometry technologies were made use of these analyses.

*2.6.3. Bioactivity attributes*

The bioactivity potential of the dried seaweeds was determined by means of a previous ethanolic (80° (v/v)) solid–liquid extraction (SLE), with a liquid–solid ratio of 30:1 (v:w) at room temperature for 60 min in an environment without light (Fernandes et al., 2021). The resulting solid was processed once again in the same conditions. The solvent of the total collected extract was removed by evaporation at 40 °C and their water fraction was eliminated by freeze-drying method. The obtained concentrated extracts were used to evaluate their antioxidant and antimicrobial properties. To sum up, their capacity to inhibit the formation of thiobarbituric acid reactive substances (TBARS), as malondialdehyde (MDA) as indicator of lipid peroxidation procedure, was tested according to Fernandes et al. (2021) technique. In addition, the cell-based oxidative hemolysis inhibition assay (OxHLIA) was also carried out to evaluate the antioxidant properties of the seaweed extract samples. This last assessment was performed as previously reported (Fernandes et al., 2021). Additionally, the antimicrobial activity was evaluated against Gram-positive bacteria (*Bacillus cereus* (food isolate), *Listeria monocytogenes* (NCTC 7973) and *Staphylococcus aureus* (ATCC 11632)) and Gram-negative bacteria (*Enterobacter cloacae* (ATCC 35030), *Escherichia coli* (ATCC 25922) and *Salmonella enterica* subsp. *enterica* serovar Typhimurium (ATCC 13311)) as well as representative micromycetes (*Aspergillus fumigatus* (ATCC 9197), *Aspergillus niger* (ATCC 6275), *Aspergillus versicolor* (ATCC 11730), *Penicillium funiculosum* (ATCC 36839), *Penicillium verrucosum* var. *cyclopium* (food isolate), *Trichoderma viride* (IAM 5061)), using Fernandes et al. (2021) methodology. Ketoconazole and bifonazole were used as positive controls. The microorganisms were purchased at the Institute for Biological Research "Siniša Stanković"- National Institute of Republic of Serbia at the University of Belgrade, Serbia.

*2.7. Recovered liquid phases characterization*

Evolution 201 UV–VIS spectrometer (Thermo Scientific, Germany) was employed for below chemical and bioactive determinations. Namely, the antioxidant capacity was evaluated by distinct colorimetric assays. Specifically, von Gadow et al. (1997) analytical method was utilized in order to determine the $\alpha$,$\alpha$-diphenyl-$\beta$-picrylhydrazyl radical (DPPH) scavenging capacity of collected liquid fractions. In this assay, samples (50 μL) were added to DPPH solution reagent (2 mL, $6 \cdot 10^{-2}$ mM) so that their absorbance was measured at 515 nm against a solvent blank after 16 min at room temperature. Conde et al. (2011) methodology was carried out for the evaluation of the ferric reducing antioxidant power (FRAP) of the different samples. In short, extracts and standard patterns (100 μL, ascorbic acid and iron sulfate heptahydrate) and work reagent (3 mL, acetate buffer 300 mM:TPTZ solution 10 mM: iron (III) chloride hexahydrate solution 20 mM (10:1:1, v/v/v)) were homogenized properly. Their absorbance value of the tested mixture was read at 593 nm after 6 min. Re et al. (1999) analysis process was done to describe the Trolox equivalent antioxidant capacity (TEAC) of the studied samples. Summarily, diluted 2′-azino-bis(3-ethylbenzothiazoline-6-sulfonic acid) diammonium salt radical cation ($ABTS^{+}$) reagent (2 mL) was reacted with sample or standard (trolox) for 6 min at 30 °C before its absorbance was measured at 734 nm.

The soluble sulfate content of these liquid samples was also assessed by the method provided by Dodgson (1961). In few words, samples (0.2 mL) were submitted at a hydrolysis process by the contact with trichloroacetic acid solution (3.8 mL, 4% (w/v)) and gelatin-barium chloride reagent (1 mL) for 15 min at room temperature. Potassium sulfate was applied for the standard curve. The absorbance was gathered at 500 nm. Khosa et al. (2011) process was used to quantity the total carotenoid content of above liquors. In this case, a SLE was development with a solid ratio of 50:1 (v/w) using *n*-hexane/acetone/absolute ethanol (2:1:1, v/v/v) solvent at room temperature for 20 min at 200 rpm in darkness conditions. The absorbance of the supernatant obtained after centrifugation at 4000 rpm for 10 min was measured at 420 nm. $\beta$-carotene was used as standard. Kamal (2017) assay allowed define the recovery liquid samples' total lipid content. For that, samples or standard (lauric acid) (500 μL) were homogenized with *n*-hexane (1500 μL) for 1 min and the mixture was read at 204 nm immediately. Singleton & Rossi (1965) procedure was followed to show the total phenolic content from each liquid sample. Briefly, aliquots (0.50 mL) were mixed with distilled water (3.75 mL), Folin-Ciocalteús reagent dissolved (1:1, v/v) (0.25 mL) and sodium carbonate solution (10%, w/v) (0.50 mL) and incubated at room temperature for 60 min in the light absence. Their absorbance was determined at 765 nm. The results were expressed as gallic acid equivalents (GAE) due to gallic acid was employed as standard pattern. Bradford (1976) assay methodology was utilized to allow

the total protein content of samples. Concisely, samples or standard (bovine serum albumin (BSA)) (1.6 mL) were vortexed with Bradford reagent (0.4 mL). After 5 min at room temperature, their absorbance value was measured at 595 nm.

Conductivity data were measured using an X5 Instrument (XS Instruments, Italy) in contrast with standard calcium chloride solutions as well as their pH values were tested in a GLP 21 pH meter (Crison, Spain).

Carbohydrate layout of these liquid samples was evaluated through HPLC instrument integrated by G1322A automatic degasser, G1311A quaternary pump, G1329A automatic injector, G1316A column furnace and G1362A refractive index detector (Agilent Technologies, Inc. Headquarters, USA). Their saccharide composition could be estimated by a previous hydrolysis treatment with sulfuric acid (4%, w/w) at 121 ± 2 °C for 40 min taking in consideration the analyze protocol reported by Balboa et al. (2013).The retention time of saccharides compounds was used to their identification as well as their quantification carried out employing standard calibration curves.

The total solid content was determined gravimetrically so that samples (1 mL) were placed in a forced air oven to dry at 105 ± 2 °C until constant weight.

Color features of seaweed defrosting liquid and aqueous phase by MHG technology were determined using CIE color space *L*a*b** above mentioned.

### 2.8. Statistical analysis

All above characterizations were performed at least in triplicate with the exception of color measurements since these carried out at least five times for each sample. Data set were showed as mean ± SD and statistically evaluation was done using one-factor analysis of variance (ANOVA) using Minitab Statistical Software® version 20.1.3.0 (Minitab, LLC, United Kingdom). A Tukey test was made to differentiate means (95% confidence, $p < 0.05$).

## 3. Results and discussion

### 3.1. Drying seaweed procedures

Fig. 2a shows the recorded extraction time, vessel temperature and collected volume values compiled during the discontinuous *C. crispus* MHG seaweed treatment. It should be highlighted that this innovative methodology permits removal of *in situ* water of tissue cells by means of microwave heating with earth gravity at atmospheric pressure (Farias et al., 2021). The first step carried out at 800 and 400 W for 2 min, respectively, whereas the second step took place at 100 W for 30 min. The used microwave irradiation power practiced a clear influence relating to the induction time necessary to recorder the first drop of collect extract since in the first phase the induction time was close to 1 min in contraposition to almost 6 min of the second phase. It is necessary to also consider the reduced moisture content and changed microstructure of the *C. crispus* matrix employed in the second stage affecting in their capacity of releases their available water fraction. The first step of MHG treatment produced a notable temperature increase until around 43 °C for the first 2 min and 93 °C at the final of this phase so the tested heating rate was ~ 17.8 °C/min. This behavior derived into a noteworthy driving force for water removal so the recovered volume was about 7 mL of extract in each intermediate step using the different irradiations powers (the flow of *in situ* liquid phase was approximately 3.5 mL/min and in both cases a linear increase was described -y = 5.95x + 20.96, $R^2$ ~ 0.92 and y = 17.18x + 25.13, $R^2$ ~ 0.96, respectively). This data was equivalent to the 56% of the total volume gathered by this procedure. The evolution of the temperature profile showed by this figure was equal to the first heating phase that characterize the MHG treatment. On the other hand, the second step of MHG procedure

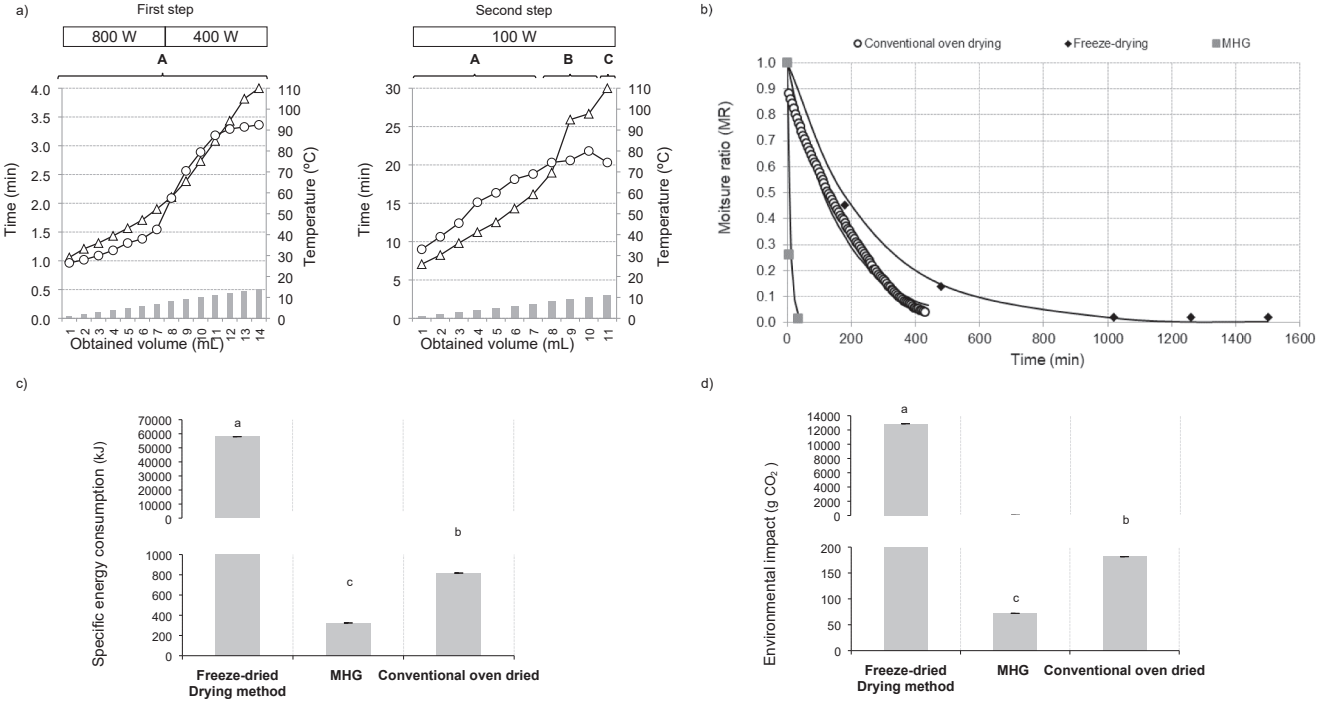


**Fig. 2.** a) Extraction kinetics of discontinuous optimized MHG treatment from *C. crispus* by the combination of different irradiation powers: first step (800 and 400 W for 2 min, respectively) and second step (100 W for 30 min). Symbols: Extraction time (triangles), vessel temperature (circles) and collected volume (bars). Typical phases of MHG treatment: heating phase (A), extracting phase (B) and burning phase (C). b) Drying kinetic of *C. crispus* seaweed by tested dried methodologies. Symbols: line (Page model). Note here that freeze-drying data (x-axis) were shortened (around 14,200 min) in order to improve data visualization. c) Estimated specific energy consumption of different drying procedures from *C. crispus* macroalgae. Data are given as mean ± standard deviation. Data values with different superscript letters are statically different ($p \leq 0.05$). d) Derived environmental impact. Data are given as mean ± standard deviation. Data values with different superscript letters are statically different ($p \leq 0.05$).

supplied the typical heating outline of complete MHG processing with a heating rate around 1.8 °C/min and a total obtained volume of 11 mL (a flow about 0.4 mL/min was described in all MHG phases emphasizing the values of the heating phase with a positive linearly of $y = 3.45x + 13.73$, $R^2 \sim 0.94$). The heating phase passed for the first 16 min whereas the extracting phase and burning phased extended for 10 min and 4 min, respectively, in order that the temperature reached a peak of 80 °C. This fact guaranteed that achieved solid phase was entire and did not show damage structure or burning features and simultaneously their moisture content (7.87 ± 0.71% d.b.) was compatible with their safe value long term preservation in accordance with other authors (López-Hortas et al., 2019b). Reached yield values statistically oscillated from 22% of freeze-dried technology to 28% of MHG procedure without significant differences with the conventional oven-dried process (about 27%). These data suggest that higher yields are linked to higher compounds retention capacity, therefore proposed MHG methodology can be selected as optimum in this technological aspect. This effect was also tested in the treatment of different vegetables as *Basella alba*, *Benincasa hispida*, *Ipomoea aquatica*, *Lagenaria siceraria*, *Musa balbisiana* Colla blossom, *Musa balbisiana* and *Musa splendida* (Mondal et al., 2019).

The kinetic curves (MR *versus* time) of drying red *C. crispus* macroalgae by the three studied methodologies is depicted in Fig. 2.b. The values indicated that conventional oven-dried treatment was the only method that facilitate a MR around 0.04 for 430 min of treatment. These samples allowed a moisture content of 9.3 ± 0.4% d.b near to the limit of an adequate dehydration condition (less to 10% d.b.). In addition, MR values in MHG and freeze-dried procedures were approximately 0.3. It should be remarked that the moisture content of freeze-dried seaweeds was ~ 4.1 ± 0.7% d.b. In the MHG case, the decrease was more remarkable since this value was achieved at 34 min in contraposition with MR value of conventional oven-dried samples at this same time (approximately 0.8). In comparison with this last methodology, the total treatment time requirement of MHG was notably shortened since this outcome was reduced around 8%. For this reason, proposed MHG drying conditions can be an effective process to water removal of *C. crispus* algae.

Dehydration kinetics, in terms of moisture ratio *vs* time, were successfully fitted to the Page model for the three studied drying methods ($R^2 > 0.94$). The following equations can be proposed for the moisture ratio variation, $MR = e^{-0.0065t}$ (conventional oven-drying), $MR = e^{-0.040t}$ (freeze-drying process) and $MR = e^{-0.1t}$ (MHG treatment). As expected, k parameter dropped from freeze to conventional drying and to MHG processing, involving a notably decrease in required processing time. The values for the conventional procedure are consistent with those previously reported for other similar carragenophyte seaweeds as *Mastocarpus stellatus* (Arufe et al., 2018).

The estimated specific energy consumption of the distinct drying procedures was defined to compare their production energy cost as represented in Fig. 2.c. MHG technology allowed a significant lower energy requirement in comparison with the others tested processing techniques. Their reduced labor energy cost (~324 kJ equivalent to 0.01 €/kWh) converted to MHG into a suitable economically procedure since their operational consume inputs kept a difference around 2.5-folds lower than conventional oven-dried system. This aspect in relation with freeze-dried method was approximately 180 times lowest due to the highest processing time and required power using this methodology. This behavior was comparable to the data literature for the drying treatment of other high moisture content matrices both at lab-scale as grapes (Farias et al., 2021). On the other hand, Fig. 2.d. exhibited the environmental impact evaluation of the three dehydrated operations. As above, MHG also supplied a significant lowest nature damage (about 72 g $CO_2$ emissions released to the atmosphere). This point indicated the MHG environmental friendly character consisting with reported research papers (López-Hortas et al., 2018).

### 3.2. Dried C. Crispus seaweed characterization

#### 3.2.1. Physical features

In order to evaluate the feasibility of the different examined drying procedures, the effect of the distinct used technologies on several physicochemical *C. crispus* macroalgae parameters were analyzed. In this context, their color features were determined instrumentally by CIE color space *L*a*b** were so that the corresponding coordinates and magnitudes are listed in Table S.1. The surface color profile of fresh seaweed was similar to the data described by this seaweed for del Olmo et al. (2020). Overall, color parameters were influenced by the used drying method in comparison with defrosted seaweed features as widely reported in the literature (López-Hortas et al., 2019b). These outputs experimented a notable reduction in all cases with significant differences with between equivalent results. This trend was verified for conventional oven-dried carried out treatment, since their color features were checked each 5 min for the complete dehydration procedure (data not shown). *L**, *a** and *b** coordinate values ranged from 15.2 for MHG samples to 21.4 for freeze-dried seaweeds, from −0.3 for MHG *C. crispus* to 0.7 for conventional oven-dried algae and from 3.1 for freeze-dried samples to 4.2 for MHG processed marine algae, respectively. Regarding the analysis of the color magnitudes, conventional oven-dried process stood out due to their higher hue-angle around 78° in contraposition with this value of freeze-dried and MHG samples (about −84° in both cases). Saturation and Chroma showed a high positive correlation coefficient (r = 0.973) with each other. This late magnitude also presented a similar relation with moisture content (r = 0.966) through the Pearson correlation analysis, likewise a similar association was displayed with *a** and *b** coordinates (r ~ 0.97 and 0.94, respectively). These results indicated that the use of color determinations can be desirable as non-destructive analytical method to determine the water content of the dried samples. The color differences between fresh seaweed and the dried *C. crispus* samples were very distinct ($\Delta E^* > 3.0$) independently of the used system according to the classification proposed by Adekunte et al. (2010). The highest total color difference data was described by freeze-dried samples (~6) consequently this methodology can turn into the degradation of their pigments as consequence of this extended treatment. A similar behavior was also described in the dry processing of *Ascophyllum nodosum* seaweed by airborne ultrasound-assisted fluidized bed drying, fluidized bed drying and oven-drying technologies (Zhu et al., 2021). The solid phase obtained for conventional oven-dried presented the lowest total color and hue differences when compared to fresh seaweed. Even though, the solid phase obtained by MHG provided the samples with highest hue difference, these seaweeds followed conventional oven-dried algae in the ranking of total color difference. For this reason, MHG can be a suitable procedure to be considered for drying treatment for *C. crispus*.

Fig. 3 records SEM images of *C. crispus* seaweed before and after tested drying methodologies. The surface morphology features of untreated and defrosted seaweeds (Fig. 3.a and b) were very similar with compact, entire, regular, and smooth microstructure so the intermedia freezing phase and posterior defrosting step carried out with a slight damage in cell wall structures only noticeable by a mild shrinkage. This phenomenon was observed more intensively in freeze-dried samples as revealed Fig. 3.c as consequence of the sublimation effect of *in situ* water during the made dehydrated treatment. Similar small-scale structure changes were previously attributed at other matrices treated at this dried procedure (López-Hortas et al., 2018). The original *C. crispus* structural arrangement still was in part exhibited in contraposition with MHG and conventional oven-dried macroalgae (Fig. 3.d and e). The photomicrographs corresponding to MHG indicated that this drying procedure was more intense at tissue level since the showed contraction increased while cell disruption was defined so that the obtained surface was irregular and without a defined shape. Above type of cell structural collapse was also displayed in the samples processed by conventional oven-dried although these matrices reported structural changes

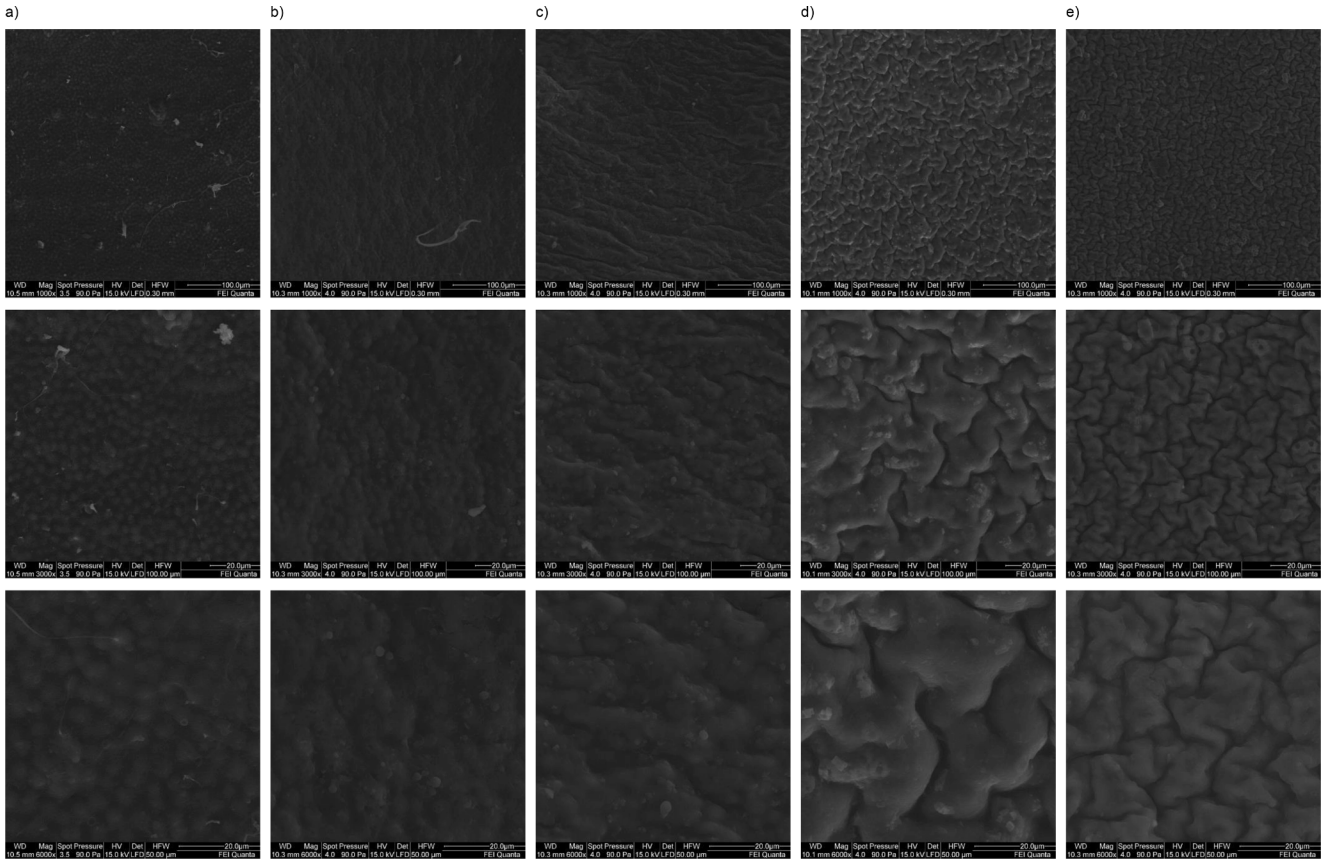


**Fig. 3.** Microscopic structure images of *C. crispus* samples untreated (a), defrosted macroalgae (b) and dried red seaweed (freeze-dried (c), MHG (d) and conventional oven dried (e)). Upper, middle and lower images with magnification of x1000, x3000 and x6000, respectively.

strongest with an evident erosion impact in their histological structure due to the intense degradation that suffered. An equivalent structural modification in the macroalgae biomass was reported by Zhu et al. (2021) at oven-drying process of *A. nodosum* seaweed.

### 3.2.2. Nutritional profile and chemical properties

The combination of environmental factors endo- or exogenous factors, as for example geographic localization, harvesting season, life stage, pH, salinity, seawater mineral levels, temperature or wave exposure, responds to the widely plasticity presented by the chemical profile of seaweeds. In particular, the nutritional values of *C. crispus* samples treated by the tested dried methods are exhibited in Table 1. Only fat content, with a range to 0.14 from 0.56 g/100 g d.b., presented significant differences between the three procedures whereas the remaining studied parameters were generally similar with each other. The average values of ash, carbohydrate and protein content were approximately, 23.8, 56.9 and 12.9 g/100 g d.b., respectively. The elemental carbon content was around 27.7 g/100 g d.b. for the distinct samples according with the contribution of this chemical element described by other red edible seaweeds as *Acanthophora spicifera* and *Gracilaria edulis* (Reka et al., 2017). This fraction can be metabolically employed in the carbohydrate storage and intercellular mucilage or can be used to the synthesis of structural polysaccharides (Lee et al., 2017). In consequence, the *C. crispus* free sugar and sulfate contents were determined. Freeze-dried and MHG biomasses did not show significant differences in relation to their soluble carbohydrate values. Specifically, their notable content can be related with the kept of the cell wall structure of algae by these systems, above mentioned, and the consequent conservation of their biochemical compounds. The fructose concentration stood out representing 89.4%, in contraposition with the 10.6% corresponding to the glucose. *Gelidium pusillum* and *Padina tetrastromatica* provided similar free sugar content so red seaweed cell walls were characterized by comparable polysaccharide profiles (Ramachandra & Hebbale, 2020). The determination of the sulfate content of dried seaweeds was outlined since sulfated polysaccharides (galactans as carrageenan oligosaccharides) exhibits gelling and thickening properties so that derived hydrocolloids show a wide range of bioactive properties. The average of obtained anion sulfate concentration was around 13.6 g/100 g d.b., which was in accordance with what earlier has been seen for and *Gelidium pacificum* (Cui et al., 2019). In relation with lipid fraction, the described reduced levels were within the expected range of Rhodophyta algae and were according with values of other red seaweeds as *Palmaria palmata* (Rajauria et al., 2015). Freeze-dried procedure produced a positive impact on the fat content data of the *C. crispus* batches since their content was about 3.5 times higher than the outputs recorded of the other drying techniques. Against this background, the preserve effect on lipid content in dry matter of freeze-drying process can be related to the preservation of the macroalgae tissues by this dried treatment and the consequent reduction of loss of lipid compounds. A similar impact has been previously highlighted to other red algae as *Kappaphycus alvarezii* (Neoh et al., 2016). This low lipid concentration also influenced in the reduced energetic contribution of the *C. crispus* matter in order that this value was similar to the average defined by others red seaweeds (Gamero-Vega et al., 2020). Data regarding the organic acids content revealed the presence of citric acid in freeze-dried *C. crispus* samples as opposed to the other matrices. This difference produced that this dried treatment presented the highest total organic content. The oxalic acid increased when freeze-dried system was used whereas the shikimic acid outcome for the three types of dried process was similar with no significance differences among them. These compounds were also defined in other edible red seaweeds like *Gigartina pistillata* and *M. stellatus* (Carpena et al., 2021).

**Table 1**
Nutritional values and composition in free sugars, organic acids, sulfate content, mineral and heavy metals of freeze-dried, MHG and conventional oven-dried seaweed *C. crispus* samples.

| Parameter | | Drying method | | |
|---|---|---|---|---|
| | | Freeze-drying | MHG | Conventional oven-drying |
| Nutritional values (g/100 g d.b.) | | | | |
| Moisture content | | 4.1 ± 0.7[b] | 7.9 ± 0.7[a] | 9.3 ± 0.4[a] |
| Ash content | | 25.8 ± 1.3[a] | 24.4 ± 0.1[a] | 21.23 ± 0.04[b] |
| Carbohydrate content | | 56.9 ± 2.5[a] | 54.6 ± 1.5[a] | 59.1 ± 3.1[a] |
| Fat content | | 0.56 ± 0.02[a] | 0.14 ± 0.01[c] | 0.18 ± 0.01[b] |
| Protein content | | 12.6 ± 0.2[a] | 13.1 ± 0.4[a] | 13.2 ± 0.5[a] |
| Energy | (kcal/100 g d.b.) | 283 ± 11[a] | 272 ± 4[a] | 291± 15[a] |
| | (kJ/100 g d.b.) | 1184 ± 46[a] | 1137 ± 18[a] | 1216 ± 61[a] |
| Free sugars (g/100 g d.b.) | | | | |
| Glucose | | 0.35 ± 0.01[a] | 0.34 ± 0.01[a] | 0.18 ± 0.01[b] |
| Fructose | | 3.0 ± 0.1[a] | 2.9 ± 0.1[a] | 2.0 ± 0.1[b] |
| Total | | 3.3 ± 0.1[a] | 3.2 ± 0.1[a] | 2.2 ± 0.1[b] |
| Sulfate content (g/100 g d.b.) | | | | |
| Total | | 14.5 ± 0.2[a] | 14.0 ± 0.3[a] | 12.1 ± 0.2[b] |
| Organic acids (mg/g d.b.) | | | | |
| Citric acid | | 22.5 ± 6.6 | nd | nd |
| Oxalic acid | | 14.6 ± 0.8[a] | 10.6 ± 0.2[b] | 10.4± 0.1[b] |
| Shikimic acid | | 0.8 ± 0.2[a] | 0.59 ± 0.04[a] | 0.69 ± 0.01[a] |
| Total | | 37.9 ± 7.6[a] | 11.2± 0.3[b] | 11.1± 0.1[b] |
| Mineral and heavy metals | | | | |
| Mineral content (mg/kg d.b.) | Na | 41,905 ± 1,759[a] | 36,369 ± 1,289[b] | 31,590 ± 1,043[c] |
| | K | 36,096 ± 1,472[a] | 33,102 ± 1,216[b] | 31,833 ± 243[b] |
| | Ca | 8,944 ± 244[a] | 4,760 ± 882[b] | 4,393 ± 84[b] |
| | Mg | 7,735 ± 245[a] | 6,930 ± 298[b] | 6,159 ± 27[c] |
| | P | 1,891 ± 1[a] | 1,671 ± 41[b] | 1,644 ± 85[b] |
| | Fe | 135 ± 9[a] | 140 ± 19[a] | 132 ± 4[a] |
| | Zn | 80.4 ± 1.9[a] | 85.1 ± 1.7[a] | 80.6 ± 2.3[a] |
| | Cu | 3.1 ± 0.1[a] | 3.3 ± 0.4[a] | 2.83 ± 0.01[a] |
| | Cr | < 2.5 | < 2.5 | < 2.5 |
| Heavy metals content (mg/kg d.b.) | I | 1,283 ± 21[a,b] | 1,387 ± 13[a] | 1,261 ± 71[b] |
| | As | 19.9 ± 0.1[a] | 18.1 ± 0.5[b] | 13.9 ± 0.2[c] |
| | Pb | 1.04 ± 0.01[a] | 0.75 ± 0.01[b] | 0.79 ± 0.11[b] |
| | Cd | 0.25 ± 0.01[c] | 0.34 ± 0.01[a] | 0.29 ± 0.01[b] |
| | Hg | 0.10 ± 0.08[a] | 0.05 ± 0.01[a] | < 0.05 |

Data are given as mean ± standard deviation except sulfate content value which standard deviation was lower than 2.5 %. Data values in a row with different superscript letters are statically different ($p \leq 0.05$). Not here that all data in the table are given in dry basis. d.b.: Dry basis; Not detected.

The prevention of malnutrition or related diseases should be supported by a high-quality intake wherein essential elements highlight. For this reason, the implementation of edible seaweeds in eating habits can facilitate the keep of a balance beneficial diet due their rife nutritional composition (Hamid et al., 2015). The mineral and heavy metals profile of *C. crispus* is also showed in Table 1. Specifically, sodium and potassium content stood out in comparison with the rest of studied compounds. Their concentrations were about 36,600 and 33700 mg/kg d.b., respectively. Their ratio near to 1 indicated that this edible marine product can be applicable to design diets for consumers with high blood pressure symptom in accordance with World Health Organization diet recommendations (Morrissey et al., 2020). Concerning calcium content, the obtained value of freeze-dried process was ~ 2-fold higher than MHG and conventional oven-dried methodologies. A similar outcome was also defined by magnesium concentration (about 6900 mg/kg d.b.) in agreement with the study of Kraan (2013). It is worth noting that in this study, that significant differences in iron, zinc and copper contents were not found among the distinct dried procedures. Their medium trace element concentrations was approximately 136, 82 and 3 mg/kg d.b., respectively. These quantities were on the same range displayed by different marine seaweed (Filippini et al., 2021; Hamid et al., 2015). The amount of chromium in studies samples was in all cases lower than 2.5 mg/kg d.b., independently of the employed dehydration procedure. These outcomes were in accordance with this content displayed previously by *Macrocystis pyrifera* (Salomone et al., 2017). On the other hand, some health hazards from anthropogenic and natural sources are associate with the presence of toxicity elements in seaweeds due to their capacity of sequester heavy-metal ions from the close environment in which they were developed. The determination of the bioaccumulation of these high persistence and toxic metal elements in the *C. crispus* tissues is necessary because of their perennial characteristic of this edible macroalga and the associated bioamplification of these potentially toxic elements along the marine food chain (Costa et al., 2020). Heavy metals in tested samples were ranked in descending order by mean values: I > As > Pb > Cd > Hg. Specifically, iodine values were in line with the literature defined previously by other dried samples (Filippini et al., 2021) considering that MHG samples recorded highest values. Their water-soluble properties affect to their concentration based on the suffered processing and cooking methods and these aspects have relevance in their deficiency or surplus contribution intakes, in relation with goiter and thyroid disorders (Wells et al., 2017). Arsenic concentration data should be monitored since the consumption of matrices with this element bioaccumulated can be related with human nervous system illness (Kraan, 2013). The freeze-dried concentration lead outcome (~1.0 mg/kg d.b.) presented significance differences with MHG and conventional oven-dried samples (about 0.77 mg/kg d.b.). In order to prevent thyroid disorders their evaluation is necessary (Alves et al., 2019), as it is also the case with iodine element above mentioned. Cadmiun average was around 0.3 mg/kg d.b. defining an amount comparable with other red algae, as *A. spicifera* and *Gracilaria corticata* (Arisekar et al., 2021) whereas mercury levels of *C. crispus* were similar to trace elements accumulated from red seaweeds (*Hypnea musciformis* and *Jania rubens* by Bonanno & Orlando-Bonaca, 2018). The exposure to these elements can impact on human health inducing carcinogenic effects and affect several human systems, as endocrine, immune, reproductive, and respiratory, among others (Suhani et al., 2021). The determination of the biosorption or bioaccumulation of the total, elemental, inorganic and organic shapes of these heavy metals as well as their mechanisms of bioaccessibility and bioavailability need to be studied further to define validated analysis protocols and limits to their monitoring and exposure associations with health risks (Bonanno & Orlando-Bonaca, 2018).

Table 2 summarizes the fatty acid profile of *C. crispus* seaweeds dried by the distinct studied procedures. Although the lipid composition of these macroalgae was limited, as shown previously in Table 1, their nutritional quality of their lipid fraction was noteworthy attributable to their widely fatty acid spectrum. The samples raised by their saturated fatty acid content, from 51.1 to 72.5% of total fatty acids defined, notifying significance differences between the dried systems. Overall, MHG samples exhibited highest concentrations with notable palmitic acid and myristic acid values about 43.3 and 3.3 %, respectively. The collected higher values of stearic acid and margaric acid were provided by conventional oven-dried procedure (around 12.2 and 1.5% in that

**Table 2**
Fatty acids composition of dried red seaweed *C. crispus* by different processed methodologies.

| Fatty acids (relative percentage, %) | Drying method | | |
|---|---|---|---|
| | Freeze-drying | MHG | Conventional oven-drying |
| C6:0 | 0.08 ± 0.01$^{c}$ | 0.51 ± 0.01$^{a}$ | 0.32 ± 0.01$^{b}$ |
| C8:0 | 0.07 ± 0.01$^{c}$ | 0.24 ± 0.01$^{b}$ | 0.27 ± 0.01$^{a}$ |
| C10:0 | 0.11 ± 0.01$^{c}$ | 0.21 ± 0.01$^{b}$ | 0.28 ± 0.01$^{a}$ |
| C11:0 | 0.02 ± 0.01$^{c}$ | 0.11 ± 0.01$^{b}$ | 0.29 ± 0.01$^{a}$ |
| C12:0 | 0.34 ± 0.01$^{c}$ | 0.69 ± 0.03$^{a}$ | 0.53 ± 0.03$^{b}$ |
| C13:0 | 0.03 ± 0.01$^{c}$ | 0.12 ± 0.01$^{a}$ | 0.09 ± 0.01$^{b}$ |
| C14:0 | 3.05 ± 0.04$^{b}$ | 3.6 ± 0.1$^{a}$ | 3.10 ± 0.04$^{b}$ |
| C14:1 | 0.09 ± 0.01 | nd | nd |
| C15:0 | 0.32 ± 0.01$^{c}$ | 0.86 ± 0.01$^{a}$ | 0.78 ± 0.01$^{b}$ |
| C16:0 | 33.6 ± 0.3$^{c}$ | 48.5 ± 0.1$^{a}$ | 47.6 ± 0.5$^{b}$ |
| C16:1 | 2.61 ± 0.02$^{a}$ | 0.86 ± 0.01$^{c}$ | 1.01 ± 0.01$^{b}$ |
| C17:0 | 0.70 ± 0.01$^{c}$ | 1.54 ± 0.01$^{b}$ | 2.16 ± 0.01$^{a}$ |
| C18:0 | 11.3 ± 0.2$^{b}$ | 11.2 ± 0.2$^{b}$ | 14.0 ± 0.4$^{a}$ |
| C18:1n9c | 39.0 ± 0.1$^{a}$ | 18.4 ± 0.2$^{c}$ | 22.9 ± 0.3$^{b}$ |
| C18:2n6c | 4.37 ± 0.01$^{a}$ | 2.67 ± 0.04$^{b}$ | 0.73 ± 0.03$^{c}$ |
| C18:3n3 | 0.23 ± 0.01 | nd | nd |
| C20:0 | 0.40 ± 0.02$^{c}$ | 0.85 ± 0.01$^{a}$ | 0.81 ± 0.01$^{b}$ |
| C20:1 | 1.25 ± 0.02$^{b}$ | 1.36 ± 0.01$^{a}$ | 1.22 ± 0.01$^{c}$ |
| C20:2 | 0.20 ± 0.01 | nd | nd |
| C20:4n6 | 0.70 ± 0.02$^{b}$ | 2.18 ± 0.02$^{a}$ | nd |
| C20:5n3 | 0.23 ± 0.01$^{b}$ | 0.66 ± 0.02$^{a}$ | nd |
| C21:0 | 0.09 ± 0.01$^{c}$ | 0.31 ± 0.01$^{a}$ | 0.27 ± 0.01$^{b}$ |
| C22:0 | 0.54 ± 0.01$^{c}$ | 2.36 ± 0.04$^{a}$ | 1.74 ± 0.01$^{b}$ |
| C22:1 | 0.09 ± 0.01$^{c}$ | 0.32 ± 0.01$^{b}$ | 0.39 ± 0.01$^{a}$ |
| C22:2 | nd | 0.71 ± 0.02$^{a}$ | 0.47 ± 0.02$^{b}$ |
| C23:0 | 0.16 ± 0.01 | nd | nd |
| C24:0 | 0.31 ± 0.01$^{c}$ | 1.34 ± 0.05$^{a}$ | 0.84 ± 0.03$^{b}$ |
| C24:1 | 0.12 ± 0.01$^{c}$ | 0.32 ± 0.01$^{a}$ | 0.22 ± 0.01$^{b}$ |
| **SFA** | **51.1 ± 0.1** | **72.5 ± 0.2** | **73.1 ± 0.2** |
| **MUFA** | **43.1 ± 0.1** | **21.3 ± 0.2** | **25.7 ± 0.3** |
| **PUFA** | **5.73 ± 0.03** | **6.21 ± 0.02** | **1.19 ± 0.05** |

Data are given as mean ± standard deviation. Data values in a line with different superscript letters are statically different ($\rho \leq 0.05$). C6:0: Caproic acid; C8:0: Caprylic acid; C10:0: Capric acid; C11:0: Undecylic acid; C12:0: Lauric acid; C13:0: Tridecylic acid; C14:0: Myristic acid; C14:1: Myristoleic acid; C15:0: Pentadecylic acid; C16:0: Palmitic acid; C16:1: Palmitoleic acid; C17:0: Margaric acid; C18:0: Stearic acid; C18:1*n*9c: Oleic acid; C18:2*n*6c: Linoleic acid; C18:3*n*3: Linolenic acid; C20:0: Arachidic acid; C20:1: Eicosenoic acid; C20:2: Eicosadienoic acid; C20:4*n*6: Arachidonic acid; C20:5*n*3: Eicosapentaenoic acid; C21:0: Heneicosylic acid; C22:0: Behenic acid; C22:1: Erucic acid; C22:2: Docosadienoic acid; C23:0: Tricosylic acid; C24:0: Lignoceric acid; C24:1: Nervonic acid; nd: Not detected; SFA: Saturated fatty acids; MUFA: Monounsaturated fatty acids; PUFA: Polyunsaturated fatty acids.

order). Freeze-dried method was the only method that displayed tricosylic acid (approximately 0.2%). These compositions were in line with other seaweeds as *Codium bursa*, *Cystoseira barbata*, *Cystoseira* compressa and *Fucus virsoides* (Cvitković et al., 2021). As regards monounsaturated fatty acids (MUFA), this moiety was ~ 30% of total fatty acid identified. Oleic acid and palmitoleic acid were the main compounds with outcomes around 26.8 and 1.5%, respectively. In this case, freeze-dried supplied the most concentrate dried samples. This variation may be due to the different localizations and harvesting period in which algae were obtained. On the other hand, polyunsaturated fatty acids (PUFA) content was approximately 4.4%, in the same rage of *G. edulis* and *Pterocladiella capillacea* based on Gamero-Vega et al. (2020) indications. In particular, linoleic acid was the highest compound delivery reporting ~ 76.3% of the total PUFA portion from freeze-dried samples following by MHG and conventional oven-dried seaweeds. The next-highest level fatty acid was arachidonic acid depicting around 35.1% when MHG methodology was employed.

### 3.2.3. Bioactivity attributes

The obtained data regarding cell-based studies antioxidant activities are compiled in Table S.2. The TBARS results of freeze-dried and MHG *C. crispus* samples showed that these dried methodologies supplied seaweed extracts capable of inhibiting the generation of MDA as secondary product of lipid peroxidation processes. The obtained average half-maximal inhibitory concentration ($IC_{50}$), namely the need extract concentration that inhibits the reaction rate to 50% of these dried treatments was around 2019 μg MDA/mL without defining significant differences among them. This value was comparable with the antioxidant capacity reported from acetone extracts of this same red macroalgae by 2,2-diphenyl-1-picrylhydrazyl assay based on Wang et al. (2009) assessment. In contrast, *C. crispus* extracts from conventional oven-dried defined the highest antioxidant activity against oxidative hemolysis of erythrocyte membranes since this sample presented the best capacity of counterattack 2,2′-azobis(2-amidinopropane) dihydrochloride (AAPH) derived peroxyl and lipophilic generated radicals. The $IC_{50}$ value in OxHLIA assay from marine alga from this last process were about 0.6 and 1.0 mg/mL at $\Delta t$ = 60 and 120 min, respectively. In comparison with the remaining systems, this represented about 1.2-fold higher antioxidant activity. Similar potential antioxidant activity results were displayed from extracts of this alga besides of *G. pistillata* and *M. stellatus* (Carpena et al., 2021). Other hydroethanolic extracts from matrices as bean fresh pods of *Phaseolus vulgaris* (Fernandes et al., 2021) also provided $IC_{50}$ values in the same range that values found in the tested dried seaweed.

The results of the antimicrobial activity of the extracts from the distinct dried *C. crispus* seaweed samples are exhibited in Table S.3. Overall, seaweed extracts presented a diverse effectiveness against the tested bacteria and/or fungal strains. In particular, the distinct *C. cripus* extracts from the evaluated drying methods showed a strong bacterial inhibitory potential since their minimum inhibition concentration values against *B. cereus*, *L. monocytogenes*, *S. aureus*, *E. cloacae* and *E. coli* were equal or lower that sodium sulfite outcomes as positive control. Regarding potassium metabisulfite as reference, dried macroalgae displayed a similar tendency except for *L. monocytogenes*, *E. cloacae* and *E. coli*, which offered higher values. MHG samples were the only ones that provided antibacterial activity in the presence of *S. typhimurium*. These data confirmed the antibacterial background knowledge of *C. crispus* as antibacterial resource since recent scientific reports revealed a similar potential against different tested species. On the other hand, studied extracts stood out since these supplied robust fungicidal inhibitory properties against all tested micromycetes when compared with the used food additives controls. In this context, *C. crispus* also presented beneficial properties against several *Candida* microorganisms (Carpena et al., 2021).

### 3.3. Recovery liquid phases characterization

MHG technique allows recovery the bioactive compounds present in the treated raw material because the produced modification of the samples structures causes the liberation and diffusion of their high value bioactive compounds through the recovery liquid fractions (López-Hortas et al., 2019a). The bioactive properties and chemical characterization of the obtained set of fractions by MHG appears described in Table 3 in comparison with the features of the collected seaweed defrosting liquid from the formulated studied proposal. Defrosted process provided around 32 mL for each 100 g of seaweed whereas the combination of the drained liquid extracts from the two steps of MHG *C. crispus* treatment offered an initial volume of reclaimed water nearly 34.8% (about 25 mL of drained liquid extracts for each 100 g of treated algae). Overall, all evaluated assays showed a similar trend so that the concentration values of seaweed defrosting liquid were significantly higher than MHG by-products. Regarding tested antioxidant capacity, defrosting phase stood out by their FRAP and TEAC features since their content was outstandingly higher than MHG extract, approximately 110 and 12 times, respectively. The total phenolic content and total carotenoid content, both parameters associated at the potential antioxidant capacity of the recovery bioactive compounds, also displayed this tendency. Results from the carbohydrate study reported that xylose and arabinose units were the predominant monosaccharides in analyzed liquid samples, representing about 93% of determined sugars. This composition was in agreement with defined seaweed polysaccharide profile previously (Laurens et al., 2020). In particular, the characterize soluble sulfate content in combination with the conductivity and pH values can be very useful for the study of *C. crispus* functional properties in addition to their seaweed sulfated polysaccharides fraction well-known. These data disclosed that extracts by MHG procedure can increase slightly stablished yields of defrosting method consequently the mixture of these aqueous phases can be favorable to improve the extraction efficiency of the studied parameters. This can be support by the analogous color coordinates and magnitudes of both types of aqueous phases (Table S.4). The described color features were comparable with literature data from MHG extracts from *Laminaria ochroleuca* and *Undaria pinnatifida* seaweeds (López-Hortas et al., 2019a and b).

**Table 3**
Antioxidant capacity and chemical composition from the *C. crispus* red algae defrosting liquid in comparison with the aqueous phase by MHG method.

| Parameter | | | Sample | |
|---|---|---|---|---|
| | | | Seaweed defrosting liquid | MHG by-product |
| Antioxidant capacity | DPPH$^{\bullet}$ (IP (%)) | | nd | 1.11 ± 0.01 |
| | FRAP | (μg ascorbic acid/g raw seaweed dry weight) | 29.51 ± 0.01$^{a}$ | 0.27 ± 0.01$^{b}$ |
| | | (μg $FeSO_4$ * $7H_2O$/g raw seaweed dry weight) | 122.37 ± 0.01 | nd |
| | TEAC (μg Trolox eq /g raw seaweed dry weight) | | 24.00 ± 0.01$^{a}$ | 1.99 ± 0.01$^{b}$ |
| Carbohydrate profile (μg/g raw seaweed dry weight) | Monosaccharides | Glucose | nd | nd |
| | | Xylose | 7.5 ± 0.1$^{a}$ | 0.4 ± 0.1$^{b}$ |
| | | Arabinose | 2.46 ± 0.01$^{a}$ | 0.10 ± 0.01$^{b}$ |
| | | Fucose | nd | nd |
| | Oligosaccharides | O-Glucose | 0.24 ± 0.03$^{a}$ | 0.003 ± 0.001$^{b}$ |
| | | O-Xylose | nd | nd |
| | | O-Arabinose | 0.1 ± 0.1$^{a}$ | 0.01 ± 0.02$^{b}$ |
| | | O-Fucose | 0.49 ± 0.03$^{a}$ | 0.02 ± 0.01$^{b}$ |
| Conductivity (mg $CaCl_2$/g raw seaweed dry weight) | | | 48.63 ± 0.01$^{a}$ | 2.1 ± 0.6$^{b}$ |
| pH | | | 5.79 ± 0.02$^{b}$ | 6.71 ± 0.02$^{a}$ |
| Soluble sulfate content (mg $SO_4^{-2}$/g raw seaweed dry weight) | | | 5.4 ± 0.1$^{a}$ | 0.27 ± 0.01$^{b}$ |
| Total carotenoid content (μg β-carotene/g raw seaweed dry weight) | | | 1.0 ± 0.1$^{a}$ | 0.04 ± 0.01$^{b}$ |
| Total lipid content (mg lauric acid/g raw seaweed dry weight) | | | 3.30 ± 0.01$^{a}$ | 0.87 ± 0.02$^{b}$ |
| Total phenolic content (μg GAE/g raw seaweed dry weight) | | | 29.66 ± 0.01$^{a}$ | 15.58 ± 0.01$^{b}$ |
| Total protein content (μg BSA/g raw seaweed dry weight) | | | 229.32 ± 0.01$^{a}$ | 18.52 ± 0.01$^{b}$ |
| Total solid content (mg extract/ g raw seaweed dry weight) | | | 82.3 ± 0.4$^{a}$ | 3.5 ± 0.5$^{b}$ |

Data are given as mean ± standard deviation. Data values in a line with different superscript letters are statically different ($p \leq 0.05$). Note here that soluble sulfate content deviations are < 2.5 %. DPPH$^{\bullet}$: 2,2-diphenyl-1-picrylhydrazyl free radical scavenging; FRAP: Ferric reducing antioxidant power; GAE: Gallic acid equivalents; IP: Inhibition percent; nd: Not detected; TEAC: Trolox equivalent antioxidant capacity.

## 4. Conclusions

To conclude, the novel method of microwave hydrodiffusion and gravity (MHG) applied to dehydrate *Chondrus crispus* seaweed offered technological advantages from the industrial point of view when compared with freeze-drying and oven-drying conventional procedures. MHG technique saved drying time (34 min), energy consumption (324 kJ) and reduced the environmental impact (72 g $CO_2$); without jeopardizing color and microstructural MHG features comparing with other traditional drying methods. The same behavior was also substantiated to the nutritive and chemical features of obtained dried seaweeds as well as their antioxidant and antimicrobial activities. MHG process permitted a remarkable additional valorization in comparison with the remaining dried methodologies since allowed to recover part of the bioactive compounds of the red macroalga by means of the aqueous extracts that can be collected during the drying procedure. This aspect can be a key factor to define an entire exploitation of the *C. crispus* seaweed at industrial level. Overall, the results obtained here could be extended to other red seaweeds, and shows MHG as an attractive alternative to dehydrate *C. crispus*. Future research should be focused on the study of the application of MHG at industrial scale in order to delve into their use as an appropriate novel dehydration technology as well as the potential applications of the recovery bioactive MHG liquid extracts in food and non-food matrices.

### *CRediT authorship contribution statement*

**L. López-Hortas:** Investigation, Methodology, Formal analysis, Visualization, Writing – original draft. **C. Caleja:** Investigation, Methodology, Formal analysis, Visualization, Writing – original draft. **J. Pinela:** Methodology, Formal analysis. **J. Petrović:** Methodology, Formal analysis. **M. Soković:** Methodology, Formal analysis. **I.C.F.R. Ferreira:** Conceptualization, Funding acquisition. **M.D. Torres:** Conceptualization, Resources, Project administration, Funding acquisition, Writing – review & editing. **H. Domínguez:** Conceptualization, Resources, Project administration, Funding acquisition, Writing – review & editing. **E. Pereira:** Investigation, Methodology, Formal analysis, Visualization, Writing – original draft. **L. Barros:** Conceptualization, Resources, Project administration, Funding acquisition, Writing – review & editing.

### Declaration of Competing Interest

The authors declare that they have no known competing financial

interests or personal relationships that could have appeared to influence the work reported in this paper.

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## Appendix A. Supplementary data

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