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Drying Kinetic Modeling and Assessment of Mineral Content, Antimicrobial Activity, and Potential α-Glucosidase Activity Inhibition of a Green Seaweed (*Ulva* spp.) Subjected to Different Drying Methods

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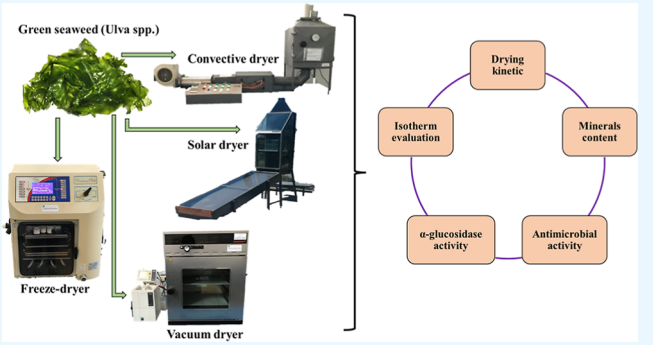
ABSTRACT: The green algal genus *Ulva* grows widely on all continents and is used for several applications such as functional foods, cosmeceuticals, nutraceuticals, and pharmaceuticals due to its nutritional characteristics. However, to increase its shelf-life and retain its bioactive components, it is necessary to apply some conservation technology, such as drying. The aim of this work is to describe the drying kinetic behavior of the green seaweed *Ulva* spp. by applying three dehydration methods: convective drying (CD), vacuum drying (VD), and solar drying (SD) by mathematical modeling and determining the retention of mineral content by atomic absorption spectroscopy and the antimicrobial potential against four strains such as *Staphylococcus aureus*, *Escherichia coli*, *Saccharomyces cerevisiae*, and *Penicillium* sp. by measurement of inhibition zones and α-glucosidase activity inhibition, as reported by IC_{50} determination. A freeze-dried sample was used as the control. The equilibrium moisture values calculated using the Guggenheim−Anderson−de Boer model were 0.0108, 0.0108, and 0.0290 g water/g d.m., for CD, VD and SD, respectively. The Midilli and Kucuk model showed robustness to fit all the experimental data of drying kinetic modeling. *Ulva* spp. is an important source of potassium with a ratio of Na/K < 0.29. Inhibition halos were observed in all samples against *S. cerevisiae* and *Penicillium* sp. with higher values than fluconazole action. An inhibitory effect on α-glucosidase activity was observed in all samples, mainly in the freeze-dried sample. Finally, dried *Ulva* spp. is a rich source of macro- and microminerals with antimicrobial activity and is a potential α-glucosidase inhibitor. Thus, it can be considered as a potential functional ingredient for food manufacturing.



1. INTRODUCTION

Seaweeds are the largest unexploited, low-trophic, and renewable global biomass resources,[1] and the green seaweed genus *Ulva* grows widely around all continents.[2] *Ulva* spp. seaweed is common along the Chilean coast,[3] with a great potential as feed for fish or mollusks,[4] as a functional ingredient,[5] and also as human food.[6] This seaweed contains a wide range of relevant components, such as polysaccharides, lipids, proteins, phenolic compounds, and pigments, and has some functional activities.[7,8] Due to its nutritional and bioactive characteristics, it is used as functional foods, cosmeceuticals, nutraceuticals, and pharmaceuticals.[9]

The mineral composition of green seaweeds has been reported to contain mainly potassium (3.1−27 mg/g dry weight), sodium (6.9−25.3 mg/g dry weight),[10] and some microminerals such as iron, zinc, copper, and manganese, which is also observed for *Ulva* spp.,[11] but their concentrations could vary based on the geographical origin and harvest season. Additionally, the fresh and lyophilized extracts from the seaweed has been described to exhibit functional properties such as anti-inflammatory activity,[12] antiviral activity,[13] antioxidant capacity,[14] antimicrobial potential,[15] and α-glucosidase inhibition capacity,[16] among others. The α-glucosidase activity is very important because it is related to type II diabetes and its inhibition may reduce carbohydrate digestion and attenuate the blood glucose levels.[17] These special characteristics generate interest in commercial exploitation for the production of a functional ingredient for food manufacturing.[18] However, to increase its shelf-life and retain

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its nutritional characteristics, bioactive components, and functional properties of the green seaweed, such as *Ulva* spp., it is necessary to apply adequate conservation technologies.

Drying is the oldest and most used industrial process to preserve foods with a higher moisture content such as fruits and vegetables, increasing their shelf-life by reducing the moisture content of the food matrix, which prevents the microbial growth.[19] This process involves heat and mass diffusion in a simultaneous process and leads to a decrease of the food volume with a decrease in the storage requirements. Thus, the use of a drying process reduces the postharvest losses, provides ease in storage and transport, and ensures product availability throughout the year.[20]

Different drying methods have been reported to analyze their effect on nutritional constituents and bioactive components either in red,[21] brown,[22] or green seaweeds.[23] Nevertheless, a detailed description of desorption isotherms and drying kinetics with mathematical modeling of the algae dehydration process is not common to find in the literature, especially for green seaweeds such as *Ulva* spp. The evaluation of the desorption isotherm by mathematical models such as the Guggenheim−Anderson−de Boer (GAB) model allows us to establish a relationship between the equilibrium moisture content and water activity at a constant temperature.[24] The use of mathematical models to describe the drying kinetic has proven to be a useful tool for the design or improvement of drying processes, allowing the prediction of water removal rates and describing the drying performance of a specific product.[25] Therefore, an in-depth description of the drying kinetic by mathematical modeling of the main drying methods used in the algae industry, that is, convection drying and solar drying (SD), is relevant for comparison with non-conventional technologies such as vacuum drying (VD) and freeze-drying (FD).

The aim of this work was to describe the behavior of the drying kinetic of the green seaweed *Ulva* spp. by applying three dehydration methods [convective drying (CD), SD, and VD] by mathematical modeling and determining the retention of the mineral content, antimicrobial activity, and potential α-glucosidase activity inhibition or using FD as the control.

2. MATERIALS AND METHODS

2.1. Raw Material. The seaweed *Ulva* spp. was collected on the coast of Guayacan, Coquimbo Region, Chile. It was cleaned and maintained in a 1000 L raceway tank (Ocean Teach S. A, Chile), where it received a constant flow of filtered seawater at a rate of 0.15 m^3/h. Upon arrival at the laboratory, the seaweed was washed with distilled water and subjected to visual inspection for the determination of size, homogeneous color, and mechanical damage. The selected fresh seaweed was dehydrated using four different drying techniques.

2.2. Drying Techniques. *Ulva* spp. was subjected to three drying techniques: CD, VD, and SD and FD which was used as a control. A convective dryer designed and built at the Department of Food Engineering at University of La Serena (La Serena, Chile) was used. Four hundred grams of the sample was placed in the dryer chamber at a load density of 2.09 kg/m^2. The hot air temperature was set at 70 °C with an airflow rate of 2.0 m/s and a relative humidity between 50 and 60%. The VD was carried out using a vacuum oven (Memmert, model VO 400, Schwabach, Germany), where 250 g of *Ulva* spp. was distributed on a stainless-steel tray at a load density of 2.07 kg/m^2 and placed inside the chamber of the vacuum dryer at 70 °C, a relative humidity between 50 and 60%, and 15 kPa. SD was carried out in a dryer designed and built at the same department as that mentioned above. This solar dryer was built with an integrated flat copper plate collector to absorb incident solar radiation and a glass sheet as a transparent cover. A thousand grams of the algae sample was spread on a stainless-steel tray at a load density of 2.06 kg/m^2, at a temperature of approximately 50 °C, with a relative humidity between 30 and 40%, and 8 h of daylight. The FD was carried out in a freeze dryer (VirTis Wizard 2.0, Advantage Plus, NY, USA). Five hundred and forty grams of the algae samples was initially frozen at −80 °C for 24 h and then quickly placed into the freeze-dryer chamber programed to be at −50 °C and 0.027 kPa for 24 h.

All drying methods were carried out until a constant weight was reached, except for FD where samples were kept in the drying chamber for 24 h. Once the samples were dehydrated, they were milled in a basic analytical grinder (IKA A-11, USA), the powdered algae were sieved using 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.

2.3. GAB Model Evaluation and Modeling of Drying Kinetic. The desorption isotherm at 50 °C was obtained from fresh green seaweed *Ulva* spp. using the method recommended by ref 26. Saturated salt solutions of LiCl, CH_3COOK, $MgCl_2$, KCO_3, Mg $(NO_3)_2$, $NaNO_3$, KI, NaCl, KCl, KNO_3, and K_2SO_4 were prepared and placed in different hermetic glass recipients. One gram of fresh seaweed was placed on a Petri dish and transferred to each recipient. Besides, a small amount of thymol was placed in each glass recipient to inhibit the microbial growth in green seaweed samples. All recipients were placed inside an oven (Memmert UF 110, Schwabach, Germany) at 50 °C. The sample weight was measured weekly using an analytical balance (±0.0001 g) (HR200, A&D Company, Tokyo, Japan) until the equilibrium of the mass was reached. Subsequently, water contents of equilibrated samples were measured by the AOAC (934.06) method in triplicate. The equilibrium moisture was calculated using the GAB model presented in eq 1.

$$X_{we} = \frac{X_m \cdot C \cdot k \cdot a_w}{(1 - k \cdot a_w) \cdot (1 + (C - 1) \cdot k \cdot a_w)} \quad (1)$$

where X_{we} [g water/g d.m. (dried matter)] is the equilibrium moisture, X_m (g water/g d.m.) represents the monolayer moisture, a_w is the water activity (dimensionless), and C and k are the constants of the model.

The weight loss values of *Ulva* spp. samples dried by CD, VD, and SD were recorded using a digital scale balance (Radwag AS 220-R2, Torunska, Poland) until a constant weight was reached, and the MR (moisture ratio, dimensionless) was determined using eq 2.

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

where X_{wt} (g water/g d.m.) is the moisture at time t, X_{we} (g water/g d.m.) is the equilibrium moisture content, and X_{w0} (g water/g d.m.) is the initial sample moisture.[27]

Eight mathematical models were used to describe the moisture loss during the drying process through predicting the MR at each drying technique. Table 1 shows eqs 3−10, which

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correspond to the mathematical models used to fit the experimental data. All models were selected from Inyang et al. (2018).[28]

Table 1. Model Equations to Drying Kinetic Description

model name	equation	equation number
Newton	$MR = \exp(-k \cdot t)$ (3)	3
Page	$MR = \exp(-k \cdot t^n)$ (4)	4
Modified Page	$MR = \exp(-[k \cdot t]^n)$ (5)	5
Henderson–Pabis	$MR = n \bullet \exp(-k \cdot t)$ (6)	6
logarithmic	$MR = a \bullet \exp(-k \cdot t) + c$ (7)	7
Weibull	$MR = \exp\left[-\left(\frac{t}{\beta}\right)^{\alpha}\right]$ (8)	8
Silva and Alii	$MR = \exp(-a \cdot t - b\sqrt{t})$ (9)	9
Midilli and Kucuk	$MR = a \cdot \exp(-k \cdot t^n) + bt$ (10)	10

The determination of each model parameter was carried out through iterative methods implemented in the free program RStudio using the dose–response analysis (drc) library[29] and the nonlinear least squares method using the brute force (nls2) library.

2.4. Extraction Method. The different dried *Ulva* spp. extracts were obtained using pure methanol and a mixture of acetone/water (70:30, v/v) as reported by García et al. (2020).[30] Dehydrated and powdered *Ulva* spp. samples were mixed with an extracting solution in a ratio of 1:10 w/v in an orbital shaker (Boeco, OS20, Germany) at room temperature and 200 rpm for 24 h. The extractions were filtered, and the filtrates were brought to dryness using a rotary evaporator (Büchi R-210, Flawil, Switzerland) at 40 °C. Then, the dry residues were resuspended in 10 mL of pure methanol or a mixture of acetone/water (70:30, v/v). Each treatment was performed in triplicate, and the products were stored at −80 °C until use.

2.5. Macro- and Microminerals. Macro- and microminerals (Ca, Na, and K and Cu, Fe, Mn, and Zn, respectively) were measured from crude ash after digestion with a mixture composed of H_2SO_4, HNO_3, and $HClO_4$ using an atomic absorption spectrophotometer (Shimadzu Instruments, Inc., SpectrAA-220, Kyoto, Japan). The mineral contents were expressed as milligrams per 100 g of dried matter and grams per 100 g of dried matter for micro- and macrominerals, respectively. All analyses were performed in triplicate.

2.6. Antimicrobial Activity. The antimicrobial activity of *Ulva* spp. extracts, reconstituted in methanol, was tested against two bacteria, one yeast, and one fungus: *Staphylococcus aureus* (ATCC 25923) (Gram-positive bacteria), *Escherichia coli* (ATCC 25922) (Gram-negative bacteria), *Saccharomyces cerevisiae,* and *Penicillium* sp., respectively. The four mentioned strains were maintained in 20% glycerol at −80 °C in a nutrient broth (Difco) at the Microbiology Laboratory, Department of Food Engineering (Universidad de La Serena, La Serena, Chile). Subsequently, the cultures were transferred to solid or liquid media. Bacterial and fungal strains were grown in 5 mL of Mueller Hinton broth (Merck KGaA, 64271 Darmstadt, Germany) under aerobic conditions for 48 h with shaking at 120 rpm at 37 °C. Then, the mother cultures were sub-cultured in tryptone soy broth (TSB, Merck KGaA, Darmstadt, Germany), incubated for 12–24 h, and used as the source of inoculums for each test. Subcultures were adjusted to a standard turbidity of 0.5 McFarland.[31]

Successively, the antimicrobial activity was evaluated using the agar diffusion method according to Kaymak et al. (2015)[32] with some modifications. 100 μL of each strain was inoculated on the surface of a Hinton Muller agar Petri plates, and sterile paper discs (6 mm in diameter) were placed. Then, 10 μL of the algae extract was impregnated on them. Ampicillin (10 μg/disk) and fluconazole (25 μg/disk) were used as the positive control and dimethyl sulfoxide (DMSO) as a negative control. The inoculated agars were incubated at 37 °C for 24 h, and the diameter of the inhibition zones was measured in millimeters.

2.7. α-Glucosidase Activity. The effect of *Ulva* spp. extracts on the α-glucosidase activity was measured in a concentration range of 0–1 mg/mL according to the procedure reported by Lordan et al. (2013),[33] with some modifications. 50 μL of the diluted extract and 100 μL of a solution of α-glucosidase from *S. cerevisiae* (Sigma G5003, Merck KGaA, Darmstadt, Germany) corresponding to 0.5 U/mL in 0.1 M sodium phosphate buffer (pH 6.9) were mixed in a 96-well microplate and incubated at 20 °C for 10 min. Subsequently, a phosphate buffer containing 50 μL of 4-nitrophenyl α-D-glucopyranoside (Sigma N1377, Merck KGaA, Darmstadt, Germany) was added to each well. The absorbance at 405 nm at 20 °C was recorded every 30 s for a total time of 10 min using a multilabel plate reader (PerkinElmer, Victor3, Turku, Finland). The α-glucosidase activity was determined as a percentage across the slope of each curve, and an exponential regression was used to fit the experimental data and determine the IC_{50}, defined as the concentration of the extract needed to produce 50% inhibition of α-glucosidase (mg/mL).

2.8. Statistical Analysis. The quality fit of the GAB model and drying kinetic mathematical models were estimated by the coefficient of determination (R^2), sum of squared errors (SSE), and chi-square (χ^2), described in eqs 11–13.

$$R^2 = 1 - \frac{\sum_{i=1}^{N} (\mathrm{Exp}_i - \mathrm{Cal}_i)^2}{\sum_{i=1}^{N} (\mathrm{Exp}_i - \overline{\mathrm{Exp}_i})^2} \quad (11)$$

$$\mathrm{SSE} = \frac{1}{N} \sum_{i=1}^{N} (\mathrm{Exp}_i - \mathrm{Cal}_i)^2 \quad (12)$$

$$\chi^2 = \frac{\sum_{i=1}^{N} (\mathrm{Exp}_i - \mathrm{Cal}_i)^2}{N - z} \quad (13)$$

where Exp_i is the experimental data, Cal_i is the calculated values, and z is the model constant numbers.[34]

The results of macro-/microminerals, antimicrobial activity, and α-glucosidase activity were expressed as the mean ± standard deviation of triplicate measurements for each analysis. The ANOVA test was performed using the free RStudio software (V. 1.4.1717) with a probability level of 5% (p = 0.05), and the multiple range test (MRT) was applied to identify homogeneous groups for all outcomes among drying methods (CD, VD, SD, and FD).

3. RESULTS AND DISCUSSION

3.1. Desorption Isotherm Evaluation and Modeling of the Drying Kinetics. A sigmoidal behavior was observed in the experimental isotherm curve (Figure 1a), exhibiting an asymptotic trend as water activity (a_w) approaches. This

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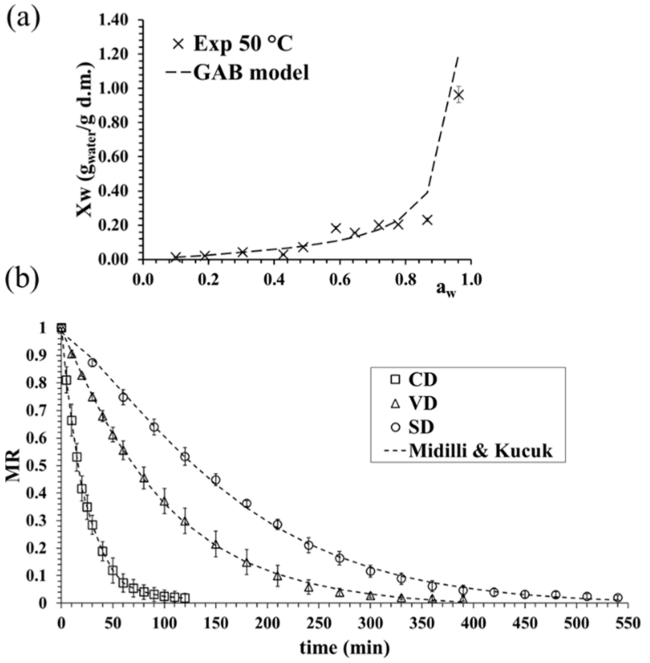

Figure 1. (a) GAB model isotherm at 50 °C and (b) drying kinetic at different dehydration techniques of *Ulva* spp. using the Midilli and Kucuk model. CD: convective drying; VD: vacuum drying; and SD: solar drying.

phenomenon has been previously described in different dehydrated marine algae such as *Mastocarpus stellatus* (red alga),[35] *Fucus vesiculosus* seaweed (brown alga),[36] *Bifurcaria bifurcata* (brown alga),[37] and *Ulva* spp. (green algae).[23] This behavior is also common in food products and is classified as a type II isotherm,[38] and it is in concordance to the desorption isotherms reported for some fruits and vegetables,[39] where it is suggested that most of the water in the fresh food matrix exerts a vapor pressure very close to that of pure water.[40] Three zones were clearly identified starting with a higher desorption area ($a_w > 0.7$), which is related to the available water for reactions and microbial growth, followed by the multilayer zone ($0.3 < a_w > 0.7$), where water molecules are less firmly attached to the food matrix, and finally the monolayer zone ($a_w < 0.3$), characterized by being constituted for more firmly bound water molecules,[40] giving it a property of unavailable and non-freezable water.[41]

The GAB equation was used to determine the equilibrium moisture from the experimental data modeling of the desorption isotherm. This model has been reported to be very useful in describing the desorption curve in some brown seaweeds such as *Sargassum muticum*[42] and *Saccharina latissima*.[43] The equilibrium moisture values calculated for CD, VD, and SD were 0.0108, 0.0108, and 0.0290 g water/g d.m, respectively. The quality fit of the GAB model was evaluated by SSE = 8.26×10^{-3}, $\chi^2 = 0.0101$, and $R^2 = 0.9646$, where the calculated values of model parameters were X_m = 0.0607 g water/g d.m., which is associated to the moisture content of the monolayer, and the C_{GAB} and k_{GAB} show values of 2.1428 and 0.9885, respectively. These mentioned GAB model parameters are related to the interaction energy between the water molecules at the individual sorption sites by the molar sorption enthalpies of the monolayer.[40]

Figure 1b depicts the MR as a function of drying time. In the three evaluated cases, a decreasing exponential curve was observed. This behavior has been reported in different drying methods[44] and agricultural products.[28]

Differences in process times with a maximum of 350% between SD and CD were observed. This is because SD depends on uncontrollable climatic factors such as day temperature and solar radiation, causing a delay in the diffusion process of water from the food matrix to the environment.

Eight mathematical models were used to describe the drying kinetic behavior of CD, VD, and SD. The model parameters are presented in Table 2.

Table 2. Parameters of Mathematical Models[a]

		drying techniques		
model		CD	VD	SD
Newton	k	0.0424 ± 0.0059	0.0102 ± 0.0010	0.0060 ± 0.0003
Page	k	0.0434 ± 0.0093	0.0071 ± 0.0001	0.0015 ± 0.0005
	n	0.9953 ± 0.0456	1.0785 ± 0.0244	1.2701 ± 0.0685
Modified Page	k	0.0425 ± 0.0060	0.0102 ± 0.0010	0.0058 ± 0.0003
	n	0.9952 ± 0.0456	1.0785 ± 0.0244	1.2701 ± 0.0685
Henderson−Pabis	k	0.0424 ± 0.0055	0.0104 ± 0.0011	0.0064 ± 0.0003
	a	1.0009 ± 0.0196	1.0171 ± 0.0065	1.0607 ± 0.0142
Logarithmic	k	0.0434 ± 0.0039	0.0097 ± 0.0014	0.0053 ± 0.0003
	c	0.0090 ± 0.0173	−0.0286 ± 0.0128	−0.0751 ± 0.011
	a	0.9958 ± 0.0143	1.0357 ± 0.0029	1.1104 ± 0.0226
Weibull	β	23.831 ± 3.2232	98.581 ± 10.3310	171.068 ± 9.814
	α	0.9953 ± 0.0457	1.0786 ± 0.0244	1.2700 ± 0.0684
Silva and Alii	a	0.0424 ± 0.0062	0.0113 ± 0.0016	0.0082 ± 0.0006
	b	−0.0002 ± 0.0240	−0.0106 ± 0.0044	−0.0289 ± 0.006
Midilli and Kucuk	a	1.0004 ± 0.0073	0.9919 ± 0.0047	0.9843 ± 0.0018
	k	0.0411 ± 0.0135	0.0071 ± 0.0011	0.0013 ± 0.0005
	n	1.0234 ± 0.0843	1.0738 ± 0.047	1.2966 ± 0.0852
	b (×10−5)	11.3170 ± 24.187	−3.2297 ± 5.231	−0.3443 ± 1.767

[a]CD: convective drying; VD: vacuum drying; and SD: solar drying. The parameters units are as follows: the drying constant k (min^{-1}), the constant models n, b, c, α, and β (dimensionless), and the shape of the materials a (dimensionless).

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Table 3. Statistical Fit for Each Drying Kinetic Model[a]

	drying techniques								
	CD			VD			SD		
model/statistics	R^2	χ^2	SSE	R^2	χ^2	SSE	R^2	χ^2	SSE
Newton	0.9971	0.000052	0.000049	0.9975	0.000221	0.000209	0.9839	0.001571	0.001489
Page	0.9974	0.000056	0.000049	0.9990	0.000072	0.000064	0.9983	0.000154	0.000138
Modified Page	0.9974	0.000056	0.000049	0.9990	0.000072	0.000064	0.9983	0.000154	0.000138
Henderson−Pabis	0.9973	0.000060	0.000049	0.9978	0.000191	0.000171	0.9836	0.001237	0.001106
Logarithmic	0.9955	0.000037	0.000030	0.9896	0.000084	0.000070	0.9938	0.000612	0.000516
Weibull	0.9974	0.000056	0.000049	0.9990	0.000072	0.000064	0.9983	0.000154	0.000138
Silva and Alii	0.9977	0.000055	0.000049	0.9986	0.000120	0.000108	0.9953	0.000473	0.000423
Midilli and Kucuk	0.9988	0.000029	0.000022	0.9994	0.000061	0.000048	0.9986	0.000148	0.000117

[a]CD: convective drying; VD: vacuum drying; and SD: solar drying.

Table 4. Content of Macro- and Microminerals in *Ulva* spp. with Different Drying Techniques[a]

	CD	VD	SD	FD
	Microminerals (mg/100 g d.m.)			
copper (Cu)	26.9 ± 2.10^b	30.40 ± 1.70^a	21.9 ± 1.50^c	34.4 ± 3.00^a
iron (Fe)	273.7 ± 7.60	300.7 ± 5.50^b	247.1 ± 13.90^d	348.3 ± 5.01^a
manganese (Mn)	36.20 ± 0.87^a	33.97 ± 0.75^a	5.67 ± 0.56^b	5.36 ± 0.27^b
zinc (Zn)	7.96 ± 2.71^b	19.90 ± 2.78^a	21.27 ± 7.17^a	20.60 ± 6.50^a
	Macrominerals (g/100 g d.m.)			
calcium (Ca)	10.85 ± 0.207^b	12.00 ± 0.436^a	9.965 ± 0.629^c	10.34 ± 0.509^c
sodium (Na)	9.01 ± 0.645^c	7.53 ± 0.474^d	15.19 ± 1.111^a	11.48 ± 0.310^b
potassium (K)	56.23 ± 0.409^a	48.19 ± 1.197^c	51.98 ± 3.427^b	54.29 ± 2.186^a

[a]Values in the same row with different superscript letters indicate significant differences ($p < 0.05$) among drying methods. CD: convective drying; VD: vacuum drying; SD: solar drying; and FD: freeze-drying.

All models demonstrated a good agreement with the drying experimental data. Therefore, according to the statistic evaluations (Table 3), the Midilli and Kucuk model obtained the best fit to all the experimental data (Figure 1b) with higher R^2 and lower values of SSE and χ^2. According to our results and the specialized literature, the Midilli and Kucuk model has shown robustness to fit the experimental data of drying kinetic modeling in various dehydration processes such as heat pump drying, solar tunnel drying, open sun drying, and microwave drying, among others.[45−48] According to Midilli et al. (2002),[49] who described this model for the first time, where the estimated parameters a and n are dimensionless constants, b is given in per minute, and k is related to the drying velocity (min^{-1}), it was observed that the parameter k increased as the process time decreased. Thus, the high k values support the elevated moisture removal rates and indicate an enhancement of the drying technology.[50]

3.2. Macro- and Microminerals. Table 4 shows the macro- and microminerals that were determined in freeze-dried, vacuum-dried, solar-dried, and convective-dried *Ulva* spp. samples. Regarding microminerals, a higher concentration of iron (Fe) was observed. This result is in concordance with that reported for *Ulva* Spp.[51] and other seaweed species such as *Codium* spp., *Halymenia floresia,* and *Saccorhiza polyschides*, where Fe is the main micromineral component.[52] Besides, a variation of Fe values between drying techniques was registered, especially in FD and VD samples, where a great retention of this element was observed. These results agree with studies on the use of vacuum in algae drying.[53]

Cupper (Cu) was also found in an important amount, especially in FD and VD samples, which statistically belong to the same homogeneous group. The high concentrations of microminerals in *Ulva* spp. could be explained by the consumption and accumulation of nutritive elements in the collection medium. The found values of manganese and zinc were higher than some results reported in the specialized literature related to *Ulva* spp. by García et al. (2016)[52] and Biancarosa et al. (2018).[54] The reason could be a seasonal and geographical difference in the green seaweeds analyzed. In addition, the use of drying methods allows a concentration of solids present in the food matrix. According to the used dehydration methodologies, VD allows a similar component retention to the FD process for manganese and an even greater retention for zinc.

Regarding *Ulva* spp. macromineral results, it is an important source of potassium with a higher concentration detected of this element in all dried samples evaluated, and similar values were reported by García et al. (2016)[52] for the same species. Sodium (Na) and calcium (Ca) were detected with similar portions between them and with slight value variations among the drying techniques applied. The ratio Na/K is very important in the human health due to the sodium intake, and the increase of the potassium intake might reduce cardiovascular events and prevent the onset of hypertension.[55] This is due to the fact that the increase in potassium absorption in the gastrointestinal tract is accompanied by a decrease in sodium absorption.[56] The potassium/sodium ratio recommended is 0.5 for an average diet.[57] According to our results, the Na/K ratios were 0.21, 0.15, 0.29, and 0.16 for dehydrated *Ulva* spp. samples obtained by FD, VD, SD, and CD, respectively. Thus, the incorporation of *Ulva* spp. in the diet could be an interesting contribution to human health.

3.3. Antimicrobial Activity. The antimicrobial activity was evaluated by the measurement of the inhibition zone on *E. coli*, *S. aureus*, *S. cerevisiae,* and *Penicillium* sp. cell layers, after the spreading in the culture medium of the extracts of *Ulva*

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Table 5. Antimicrobial Activity of Fresh and Dried *Ulva* spp. by CD, VD, SD, and FD against Some Microorganisms[a]

	inhibition zone (mm)			
Ulva spp.	*E. coli*	*S. aureus*	*S. cerevisiae*	*Penicillium* sp.
fresh	6.00 ± 0.08[d]	—	8.77 ± 0.85[c]	12.87 ± 0.59[b]
CD	—	—	10.66 ± 0.58[b]	11.25 ± 0.96[bc]
VD	7.15 ± 0.31[c]	—	10.47 ± 0.50[b]	11.40 ± 1.81[bc]
SD	—	—	9.30 ± 0.01[c]	10.60 ± 1.05[c]
FD	8.98 ± 0.50[b]	7.55 ± 0.17[b]	11.97 ± 0.15[a]	15.87 ± 0.35[a]
negative control[b]	0	0	0	0
positive control[c]	16.05[x] ± 0.04[a]	15.32[x] ± 0.06[a]	8.96[y] ± 0.06[c]	9.95[y] ± 0.07[c]

[a]Values are expressed as mean ± standard deviation. Different letters in the same column indicate significant differences ($p < 0.05$) according to the MRT. Standard deviation was calculated on three replicates. (—) no visible zone. [b]Positive control: ampicillin (10 μg/disk) or fluconazole (25 μg/disk) for bacteria and fungi, respectively. [c]Negative control: DMSO. CD: convective drying; VD: vacuum drying; SD: solar drying; and FD: freeze-drying.

spp. reconstituted on pure methanol. Table 5 shows the dimension of the inhibition zone diameter (mm) for each dried sample. Inhibition halos were observed in all samples against *S. cerevisiae* and specially on *Penicillium* sp., where the values registered for *Ulva* species were in accordance with the results reported by Abdel-Khaliq et al. (2014).[58] A great inhibition zone in the extracts of the FD sample compared to that in the fresh extract was observed, and all evaluated samples showed a greater halo diameter than the positive control (fluconazole). However, significant differences were observed among the drying methods, with the FD process followed by VD being the only ones that allow a major retention of the antimicrobial activity on *S. cerevisiae* and *Penicillium* sp. This result is an interesting finding, indicating the potential of the extract of *Ulva* spp. as an antifungal. Studies on *Ulva lactuca* (a green alga) extracts have shown to affect fungal growth and their activity,[59] generating an important precedent that validates our results.

On the other hand, both *E. coli* and *S. aureus* bacteria have been shown to be more resistant, with inhibition halos observed in FD, VD, and fresh samples for *E. coli* and FD samples for *S. aureus*. However, the found values are similar to those reported in the specialized literature.[60] The diameters of halos were smaller than that of the positive control (ampicillin), with an average difference of about 50%. Nevertheless, the dried extracts had a better antimicrobial effect than the fresh extract. Therefore, according to our results, the drying applied methods improved the antimicrobial action against the different evaluated strains probably through bioactive component concentration.

3.4. Potential of α-Glucosidase Inhibition. The reconstituted dried extracts on acetone/water solution in a 70:30 proportion were used at different concentrations to evaluate the α-glucosidase activity. The ability to inhibit the activity of this enzyme is an important system for regulating glucose absorption and could have a positive effect in the control of type II diabetes.[61] Figure 2 shows the IC_{50} values, where all samples presented an inhibitory effect on α-glucosidase activity. The type of drying method conditioned the effect of dried extracts of *Ulva* spp. on the ability to inhibit the enzyme α-glucosidase, observing different homogeneous groups. The FD method is the one that proved to be the most effective since a concentration of 0.408 ± 0.017 mg/mL is needed to reduce the enzymatic activity by half. Although the samples dehydrated by VD, CD, and SD have an inhibitory effect, a higher concentration is needed to decrease the α-glucosidase activity, registering values of 0.507 ± 0.058, 0.508 ± 0.02, and 0.597 ± 0.018 mg/mL, respectively. Previously published results by our working group showed the presence of phenols and flavonoids in *Ulva* spp.,[23] which could be related to their inhibitory capacity toward α-glucosidase activity.[62]

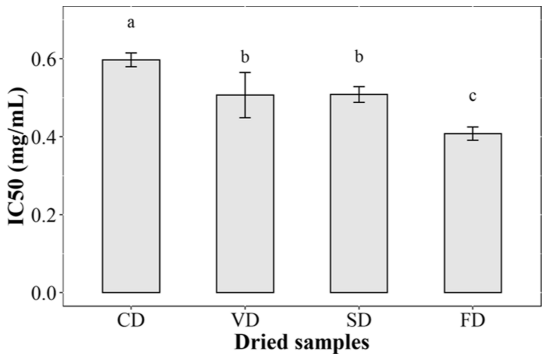

Figure 2. IC_{50} (half-maximal inhibitory concentration) values of *Ulva* spp. extracts dried by FD, SD, CD, and VD techniques. Bars with the same lowercase letters are not significantly different ($p < 0.05$). Values are averages ($n = 3$), and error bars are standard deviation. CD: convective drying; VD: vacuum drying; SD: solar drying; and FD: freeze-drying.

These findings indicate that the inhibition of α-glycosidase by *Ulva* spp. extracts could generate interest in future studies for a pharmacological approach in this alga toward a treatment against diabetes.

4. CONCLUSIONS

The drying kinetics of *Ulva* spp. was evaluated considering three conventional drying technologies. Subsequently, the macro- and micromineral contents were determined, and the antimicrobial effect of dried extracts was evaluated as well as their ability to inhibit the enzymatic action of α-glucosidase, and the freeze-dried *Ulva* spp. extract was used as the control. According to the results, we present the following conclusions:

- The desorption isotherm curve of *Ulva* spp. was classified as a type II isotherm and described in detail by the GAB model, allowing the determination of the equilibrium moisture, which varies between 0.0108 and 0.0290 g water/g d.m. depending on the drying technique applied.
- The drying kinetic was predicted with the best fit using the Midilli and Kucuk model in all the experiments carried out, registering a typical decrease exponential curve. The CD was the fastest drying process, and this

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was validated by a higher value of the *k* parameter of the Midilli and Kucuk model.

- *Ulva* spp. showed a relevant content of macro- and microminerals and specially presented a low ratio of Na/K, being <0.29, which is important in diet for the decrease of sodium absorption.
- The FD extract (drying method control) showed a great antimicrobial activity, mainly on *S. cerevisiae* and *Penicillium* sp., exhibiting a better antimicrobial effect than that of the fresh extract. Therefore, the drying process concentrated and preserved the components that generated this activity. On the other hand, a relevant α-glucosidase activity inhibition was obtained in all evaluated extracts, and these findings could generate interest in future studies for a pharmacological approach for green seaweeds against diabetes.
- VD becomes important because it allows obtaining a better retention of chemical components and biological activities similar to that obtained using the control method (FD). Therefore, it could be considered as a non-conventional food drying alternative to the FD process.
- Finally, the dried *Ulva* spp. is a rich source of macro- and microminerals, having antimicrobial activity and α-glucosidase inhibition activity and being a potential functional ingredient for food manufacturing.

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Notes

The authors declare no competing financial interest.

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