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Drying method matters: Differential preservation of lipids, pigments, and carbohydrates in edible macroalgae *Fucus vesiculosus*, *Gracilaria* sp., and *Ulva* sp.

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ABSTRACT

Marine macroalgae are increasingly recognized as sustainable sources for food, feed, and functional applications. Drying is widely used to stabilize biomass and preserve nutrients and bioactive compounds. Freeze-drying (FD) effectively preserves sensitive compounds, but high cost and energy demand limit scalability. Therefore, this study evaluated air-tunnel drying (ATD; 25 °C) and solar drying (SD; 40 °C) as alternative methods for preserving lipid, fatty acid (FA), pigment, and carbohydrate composition of edible *Fucus vesiculosus*, *Gracilaria* sp., and *Ulva* sp., using FD as a reference. In most cases, ATD preserved lipid and carbohydrate contents comparable to FD. In *Ulva* sp., ATD promoted hydrolytic FA release, increasing free FA, while ulvan polysaccharides remained stable across drying methods. *F. vesiculosus* exhibited high stability, maintaining lipid content, FA composition, and

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pigment levels under all conditions. SD induced species-dependent alterations, including reductions in lipid content, polyunsaturated FA, and pigment levels in *Gracilaria* sp. and *Ulva* sp. Although *F. vesiculosus* polysaccharides remained largely unchanged, a potential increase in fucoidans was observed under SD, suggesting a stress-induced response. SD also decreased galactose and sulphate esters contents in *Gracilaria* sp. These findings support the selection of drying strategies according to species, target compounds and applications to optimize biomass quality.

1. Introduction

Marine macroalgae (seaweeds) have attracted increasing interest as sustainable biomass resources with diverse nutritional composition, positioning them as promising ingredients for food and feed applications (Fasogbon et al., 2024; Matos et al., 2024; Sumana et al., 2026). They provide a wide range of compounds, including lipids, carbohydrates, and photosynthetic pigments, which collectively contribute to their nutritional value and associated health benefits (Lomartire et al., 2021).

Macroalgae are recognized as valuable sources of lipids, particularly omega−3 and omega−6 polyunsaturated fatty acids (PUFA). Although lipids generally account for less than 10% of their biomass, this fraction is nutritionally important due to the presence of compounds, such as α-linolenic acid (18:3 *n*−3, ALA), linoleic acid (18:2 *n*−6, LA), eicosapentaenoic acid (20:5 *n*−3, EPA) and arachidonic acid (20:4 *n*−6, ARA), which are associated with immune regulation and inflammatory processes, acting as regulators of the circulatory system (Marques et al., 2021; Seth et al., 2024). In addition, macroalgae are particularly rich in carbohydrates, comprising structural polysaccharides that largely contribute to dietary fiber intake (Huang et al., 2022; Matos et al., 2024). Sulphated polysaccharides (e.g., fucoidans found in brown macroalgae and ulvans found in green macroalgae) are widely recognized for their antioxidant, anticoagulant, antiviral and immunomodulatory effects, among others (Kidgell et al., 2019; Lomartire and Gonçalves, 2022; Nguyen et al., 2024). Due to their limited digestibility in the upper gastrointestinal tract, these polysaccharides reach the colon where they are partially or fully fermented, acting as dietary fiber and supporting gut health, glycemic regulation and protection against chronic diseases (Lordan et al., 2011; Wells et al., 2017; Huang et al., 2022). Macroalgae also contain a diverse array of photosynthetic pigments, including chlorophylls, carotenoids (e.g. fucoxanthin), and phycobiliproteins, which exhibit antioxidant activity and functional properties of interest for food applications. These natural pigments are widely used as food colorants, enhancing the sensorial and techno-functional properties of food products. Their application has been extended to intelligent food packaging systems to monitor food freshness and act as time-temperature indicators, thereby contributing to improved food quality and shelf-life (Manzoor et al., 2024). However, the effective exploitation of these high-value compounds strongly depends on appropriate post-harvest handling and preservation of the algal biomass. Pigments, for instance, are particularly susceptible to degradation under typical processing and storage conditions (Cikoš et al., 2022). In this context, conservation strategies are a key factor in maintaining biomass quality and ensuring the stability of nutritionally and functionally relevant compounds.

Fresh macroalgal biomass poses significant challenges, as its high water content (>90%) enables microbial and enzymatic activity, making the fresh biomass highly perishable (del Olmo et al., 2020). Consequently, drying represents a crucial post-harvest processing step to stabilize macroalgal biomass, decrease water activity, and extend shelf-life, while preserving biochemical integrity (Gupta et al., 2011; Oliveira et al., 2020; Zhu et al., 2021). However, drying processes may also induce structural and chemical modifications that affect the integrity and stability of the compounds (Healy et al., 2023). Temperature, drying time, water content, light, and oxygen exposure are parameters that can promote the loss of the compounds found in macroalgae, including pigments and oxidation-sensitive lipids (Dhakal et al., 2024; Nunes et al., 2024). Although relatively resistant, seaweed carbohydrates can be affected by drying methods, which may influence their stability, structural integrity, and extractability. As a result, the choice of drying technologies plays a decisive role in determining the nutritional and functional attributes of dried macroalgal biomass.

Among the variety of drying methods currently applied in macroalgal processing, freeze-drying (FD) is often considered the benchmark technique due to its effectiveness in preserving thermolabile and oxidation-sensitive compounds. However, its high energy demand and associated costs limit large-scale industrial implementation (Santhoshkumar et al., 2023). Convective drying methods, including air-drying, are widely used in food processing due to their operational simplicity and relatively low costs (Chen & Pan, 2023; Asrate & Ali, 2025). These techniques rely on forced convection to remove moisture, causing water to evaporate into the surrounding air. As moisture content decreases, water activity is reduced, resulting in enhanced shelf-life (Obadi et al., 2023; Asrate & Ali, 2025). Despite these advantages, convective drying may induce structural changes in the food matrix, potentially affecting product quality and sensory attributes (Augustin et al., 2016). Nevertheless, air-drying remains an attractive option due to its comparatively high energy efficiency and scalability (Villagracia & Ong, 2022; Hernández et al., 2024). When appropriately controlled, it can mitigate heat-induced degradation of sensitive compounds, contributing to the preservation of nutritional and bioactive compounds (Aljabri et al., 2023).

Solar drying systems, represent a more sustainable and cost-effective alternative, as they rely on renewable solar energy and minimize the need for external power sources (Lingayat et al., 2020; Silva et al., 2021; Schmid et al., 2022). However, direct exposure of biomass to solar radiation may degrade sensitive compounds and loss of functional properties, particularly pigments and lipids (Uribe et al., 2019; Ullah et al., 2025). To address these limitations, indirect solar drying systems have been developed, in which solar energy is used to heat air that is subsequently circulated through the biomass, thereby maintaining the quality of biomass (Mohammed et al., 2020; Schmid et al., 2022). Further process stability can be achieved using hybrid solar dryers that combine solar irradiation with auxiliary components such as fan heaters, dehumidifiers, and controlled ventilation. These systems ensure stable temperature and humidity conditions under variable climatic conditions, while maintaining low overall energy consumption.

Previous studies have demonstrated that drying methods can significantly influence the biochemical composition of macroalgae (Badmus et al., 2019; Regal et al., 2020; Uribe et al., 2020; Lopez-Hortas et al., 2022; Zhao et al., 2022; Tolstorebrov et al., 2024). However, the reported effects are often inconsistent, with both losses and retention of compounds depending on algae species and drying conditions. Studies that directly evaluate different drying methods comparing macroalgae belonging to different phyla remain scarce. Furthermore, even among species belonging to the same genus (e.g., *Fucus*), contrasting results have been reported under similar drying conditions, particularly regarding the lipid fraction (Badmus et al., 2019; Dhakal et al., 2024). This gap highlights the need for comparative studies evaluating the effects of drying methods on macroalgae with distinct biochemical compositions and taxonomic backgrounds. As a general trend, fatty acids (FA), particularly PUFA, are susceptible to oxidative degradation during hot-air drying, with enhanced oxidation observed at moderate temperatures (40–60 °C) (Badmus et al., 2019; Regal et al., 2020; Lopez-Hortas

et al., 2022; Wirenfeldt et al., 2024), although the extent of degradation varies among species. Pigments are highly sensitive to thermal- and light-induced stress (Uribe et al., 2020; Zhao et al., 2022). In contrast, the carbohydrates generally exhibit greater thermal stability (Stévant et al., 2018; Silva et al., 2019), but drying conditions may still affect their content and availability through depolymerization, structural rearrangements or enhanced extractability (Silva et al., 2019; Montes et al., 2021) or even *de novo* synthesis under stress conditions (Oliveira et al., 2009; Cardoso et al., 2010).

In this context, the present study aimed to evaluate the impact of three drying methods on nutritionally relevant compounds in macroalgae. Freeze-drying (FD) was used as a reference technique, as the most adequate to preserve the composition of biomass, while air-tunnel drying (ATD) and solar drying (SD) were selected as industrially relevant representative methods. The effects of these drying strategies were assessed on lipid content and FA composition, as well as pigment and carbohydrate profiles. Three edible macroalgal species widely used in food applications and representative of the three phyla were investigated: *Fucus vesiculosus* (Ochrophyta), *Gracilaria* sp. (Rhodophyta) and *Ulva* sp. (Chlorophyta). This study provides valuable insights into optimizing macroalgal drying strategies for food and bioprocessing applications.

2. Material and methods

2.1. Macroalgae samples and drying methods

The macroalgae species *F. vesiculosus*, *Gracilaria* sp. and *Ulva* sp. were produced by ALGAplus, SA at a production site located at Ria de Aveiro coastal lagoon, Northern Portugal (40°36′43″ N, 8°40′43″ W), using an open land-based integrated multi-trophic aquaculture (IMTA) system. The biomass was cultivated under standardized organic production conditions routinely applied at ALGAplus, using filtered seawater from the Ria de Aveiro. To minimize possible differences in macroalgae biochemical composition arising from growth conditions, harvest stage, nutrient availability, and seasonal variability, all samples used in this study were harvested on the same day, at the end of the production cycle (October 2024). Although the same general production system, harvest period, and post-harvest workflow were used for all species, stocking density was adjusted according to each macroalgal species. After harvesting, the biomasses were washed with filtered and sterilized seawater. Fresh samples were then submitted to three different drying methods: freeze-drying (FD), air-tunnel drying (ATD) and solar drying (SD). Freeze-drying was performed by initially freezing the samples at −80 °C, followed by lyophilization in a LyoMicron freeze dryer (Coolvacuum, Spain) operating at an absolute pressure of 0.04 mbar and a condenser temperature of −70 °C, during 4 days. Air-tunnel drying was carried out by arranging the samples on trays and drying them in an industrial convective forced air-tunnel drier (customized built by ALGAplus SA) at 25 °C. Drying was performed until the samples reached a moisture content below 12%, resulting in drying times ranging from 10 to 22 h depending on the species. Moisture content was determined using a ML−50 Moisture Analyzer at 2 h intervals after the first 10 h of drying. Variations in drying time among species depended on differences in morphological and structural characteristics, including thallus thickness, surface-to-volume ratio, and initial moisture content. Solar drying was conducted at Allmicroalgae S.A. using an industrial indirect hybrid solar dryer (BBKW Drying Solutions, Portugal), in which samples were placed on trays and convectively dried for 22 h at a maximum temperature of 40 °C and a minimum relative humidity threshold of 25%, according to Van De Walle et al. (2024) and Baune et al. (2025). The system operates primarily using solar energy, where ambient air is heated by solar irradiance and subsequently circulated through the drying chamber, thereby avoiding direct exposure of the biomass to sunlight. Airflow within the dryer is mechanically assisted by a set of fans (TVM 24 D; 124 W, Trotec GmbH, Heinsberg, Germany) to ensure homogeneous drying. Drying conditions were controlled by a software-based algorithm that uses temperature and humidity sensors to regulate air intake and exhaust, as well as a dehumidifier (TTK 350 S; 70 L/ 24 h, 1.3 kW, Trotec GmbH, Heinsberg, Germany) and a fan heater (TDS 20; 3.3 kW, Trotec GmbH, Heinsberg, Germany). Detailed characteristics and operating conditions of the solar dryer are described by Schmid et al. (2022).

2.2. Determination of moisture content and moisture correction factor

Moisture content of dried biomasses obtained using the three different drying methods was determined by drying the samples (100 mg) for 15 h at 105 °C in a drying chamber (model no. 52301–45, Cole-Parmer Instrument Co., Chicago, IL, USA). The crucibles were cooled to room temperature in a desiccator and weighed to determine moisture content by gravimetry (Moreira et al., 2025). All analytical results obtained from dried samples (except their moisture content) were expressed to a dry weight (DW) basis using moisture correction factors (MCF), calculated as MCF = 100 / (100 − residual moisture content, g 100 g^{-1}).

2.3. Lipid extraction

Lipids of *F. vesiculosus*, *Gracilaria* sp., and *Ulva* sp. were extracted from 250 mg of dried biomass using a modified Bligh and Dyer method (Lopes et al., 2020). The biomass was mixed with 5 mL of methanol (MeOH):dichloromethane (DCM) (1:1, v/v), vortexed, and incubated on ice for 30 min. After centrifugation at 2000 rpm for 5 min at 4 °C, the organic phase was collected, and the process was repeated two times. The extract was dried under nitrogen and re-dissolved in 4 mL of DCM: MeOH (1:1, v/v). Phase separation was achieved by adding 1.8 mL of Milli-Q water, vortexing, and centrifuging. The organic phase was then collected and the aqueous phase re-extracted with 2 mL of DCM. Combined organic phases were filtered, dried under nitrogen, transferred to pre-weighed amber vials, and stored at −20 °C. Lipid content was determined gravimetrically as a percentage of dried biomass.

2.4. Fatty acid composition analysis

2.4.1. Fatty acid derivatization

Fatty acids methyl esters (FAME) were prepared from 30 μg of lipid extracts of *F. vesiculosus, Gracilaria* sp., and *Ulva* sp., using two derivatization approaches. Alkaline-catalyzed methylation with KOH was applied to quantify esterified FA, as described by Monteiro et al. (2022), while acid-catalyzed methylation with acetyl chloride was used to analyze the total FA profile (esterified and non-esterified FA), according to Vizetto-Duarte et al. (2019).

For the alkaline derivatization, lipid extracts were combined with 1 mL of internal standard solution (methyl nonadecanoate in 99% *n*-hexane, 1 μg mL^{-1}), followed by 200 μL of KOH solution (in MeOH, 2 M), and vortexed. For the acetyl chloride derivatization, lipid extracts were mixed with 1.5 mL of acetyl chloride:MeOH solution (1:20, v/v), vortexed, and 1 mL of the same internal standard solution was added to each sample, followed by incubation for 1 h at 80 °C in a heating block. Phase separation was induced by adding 2 mL of saturated NaCl solution (10 mg mL^{-1}) for alkaline conditions and 1 mL of Milli-Q water for acetyl chloride conditions, followed by centrifugation at 2000 rpm for 5 min at 4 °C. Then, the organic phase was collected, dried under nitrogen, and dissolved in 100 μL of 99% *n*-hexane for gas chromatography–mass spectrometry (GC-MS) analysis.

2.4.2. Fatty acid analysis by GC-MS

Fatty acids methyl esters were analyzed by GC–MS, as previously described in Monteiro et al. (2022). Briefly, 2 μL of the prepared FAME solution was injected into a GC-MS equipped with a DB-FFAP column. The injector and detector temperatures were set at 220 °C and 230 °C,

respectively. The oven temperature followed a programmed gradient, starting at 58 °C (held for 2 min), increasing to 160 °C at a rate of 25 °C min^{-1}, then to 210 °C at 2 °C min^{-1}, and finally reaching 225 °C at 20 °C min^{-1}. The system operated in electron impact mode with helium as the carrier gas. FAME were identified by comparing their retention times and mass spectra with standard references (Supelco 37 Component FAME Mix) and databases (NIST Library, "Lipid Web"). Results were processed using Agilent MassHunter software.

Based on the FA contents, the atherogenic (AI) and thrombogenic (TI) indices were calculated using the following formulas, according to Ulbricht and Southgate (1991):

$$AI = \frac{[C12:0 + (4 \times C14:0) + C16:0]}{\Sigma MUFA + \Sigma n-6 + \Sigma n-3} \quad (1)$$

$$TI = \frac{C14:0 + C16:0 + C18:0}{[(0.5 \times \Sigma MUFA) + (0.5 \times \Sigma n-6) + (3 \times \Sigma n-3) + (\Sigma n-3/\Sigma n-6)]} \quad (2)$$

2.5. Carotenoids and chlorophyll estimation

Total carotenoids and chlorophyll contents were estimated spectrophotometrically (Pinho et al., 2025). Briefly, aliquots of 50 μg of the lipid extracts of *F. vesiculosus*, *Gracilaria* sp., and *Ulva* sp. were combined with 500 μL of MeOH. The absorbance of 200 μL of each sample was measured at 450 nm and 670 nm for carotenoids and chlorophylls quantification, respectively, with a microplate reader (Multiskan™ GO 1.00.38, Thermo Scientific, Hudson, NH, USA). The total concentrations of carotenoids and chlorophylls were estimated based on commercial pigments standard curves, fucoxanthin (0–40 μg mL^{-1}) and chlorophyll *a* (0–100 μg mL^{-1}), respectively.

2.6. Chemical composition analysis of carbohydrates

2.6.1. Neutral sugars analysis

Neutral sugars were analyzed using gas chromatography-flame ionization detection (GC-FID) after being converted into their respective alditol acetates, following the method described by Ferreira et al. (2020). Samples (1–2 mg) were pre-hydrolyzed in 200 μL of 72% (w/w) H_2SO_4 for 3 h at room temperature and were vortexed occasionally. The pre-hydrolysis was followed by a hydrolysis in 2.4 mL of 1 M H_2SO_4 at 100 °C for 2.5 h. A volume of 200 μL of 2-deoxyglucose solution (1 mg mL^{-1}) was used as internal standard. The released monosaccharides were then reduced with 15% (w/v) $NaBH_4$ prepared in 3 M NH_3 for 1 h at 30 °C and acetylated with acetic anhydride for 30 min at 30 °C, using methyl-imidazole as a catalyst, to produce alditol acetates. The 3,6-anhydrogalactose (3,6-AnGal) sugar residue was determined through reductive hydrolysis (Stevenson & Furneaux, 1991; Vinagre et al., 2025). This procedure involves hydrolysis with 3 M trifluoroacetic acid and simultaneous reduction with 4-methylmorpholine-borane (80 mg mL^{-1}), followed by acetylation with acetic anhydride to yield alditol acetates. Mannitol of *F. vesiculosus* was also analyzed by its conversion into mannitol acetate by acetylation, using 2-deoxyglucitol (1.0 mg mL^{-1}) as internal standard. The alditol acetates were analyzed using a Perkin Elmer Clarus 400 GC-FID. The injector and detector were operated at temperatures of 220 °C and 240 °C, respectively. The oven temperature program began at 220 °C, held for 7 min, and then increased to 240 °C at a rate of 10 °C min^{-1} . Hydrogen was used as the carrier gas, with a flow rate of 1.7 mL min^{-1} .

2.6.2. Uronic acids content

Uronic acids (UA) were quantified using the *m*-phenylphenol colorimetric method (Lopes et al., 2026). Following pre-hydrolysis (200 μL of 72% H_2SO_4 for 3 h at room temperature), the samples were hydrolyzed in 2.4 mL of 1 M H_2SO_4 for 1 h at 100 °C. The hydrolyzed samples were diluted (1:3, v/v), and 100 μL of each sample was added to 1 mL of 200 mM boric acid prepared in 98% (w/w) H_2SO_4. The mixture was stirred and heated at 100 °C for 10 min. After cooling down on ice, 20 μL of *m*-phenylphenol were added and the samples were transferred to a microplate for absorbance measurement at 520 nm using a BioTek - Eon Microplate Reader. The total amount of uronic acids was determined based on a standard curve of D-galacturonic acid (0–100 μg mL^{-1}), and the results were expressed as GalA equivalents.

2.6.3. Sulphate content

The content of total sulphates in macroalgae biomasses was determined by the turbidimetric method (Dodgson, 1961; Dodgson & Price, 1962). Barium chloride–gelatine reagent was prepared in advance by dissolving 1 g of gelatine in 200 mL of hot water (60–70 °C) and allowing the solution to stand overnight at 4 °C. Barium chloride (0.75 g) was then dissolved in the semi-gelatinous fluid, resulting in a cloudy solution that was left to stand for 2–3 h prior to use. Samples were accurately weighed (4–5 mg) and dissolved in 1 mL of 1 M HCl, followed by a hydrolysis at 105–110 °C for 5 h. After being cooled, an aliquot of 100 μL of the hydrolysate was then transferred to a glass tube containing 1.9 mL of 3% (w/v) trichloroacetic acid. Subsequently, 0.5 mL of barium chloride–gelatine reagent was added and the mixture was thoroughly vortexed and allowed to stand at room temperature for 15–20 min. After 15–20 min, the absorbance of the stabilized suspension of barium sulphate was measured at 360 nm in quartz cells (microplate reader Eon, Biotek, USA) against a reagent blank prepared using distilled water in place of the hydrolyzed sample. A second 100 μL aliquot of the hydrolysate was combined with 1.9 mL of trichloroacetic acid, as described above, and with 0.5 mL of gelatine solution (i.e., with no barium chloride). The absorbance of this "control" solution was also measured using 360 nm in quartz cells against a reagent blank prepared with distilled water in place of the hydrolysate. This control provided a measure of the ultraviolet absorbing materials formed during hydrolysis, and the corresponding absorbance value was subtracted from that of the test solution. Sulphate concentrations were quantified using a calibration curve built with potassium sulphate (K_2SO_4), corresponding to 10–100 μg of SO_4^{2-} ions.

The free sulphates, *i.e.*, sulphates that are not covalently linked to carbohydrates, were also determined by the turbidimetric method described above, but without performing the 5 h hydrolysis step. The sulphate esters of the carbohydrates were determined by subtracting the content of free sulphates to the total sulphates content assessed for the different macroalgae.

2.7. Statistical analysis

All analyses were performed in triplicate, with results presented as mean ± SD ($n = 3$). Statistical analysis was performed using GraphPad Prism version 8.0.1 for Windows (GraphPad Software, San Diego, CA, USA). Data normality was assessed using the Shapiro-Wilk test. One-way ANOVA followed by Tukey's post hoc test was used to compare differences among drying methods. For non-normally distributed data, the Kruskal-Wallis test followed by Dunn's post hoc test was applied.

Table 1
Moisture content (expressed as g 100 g^{-1} dried biomass) of *Fucus vesiculosus, Gracilaria* sp. and *Ulva* sp. subjected to freeze-drying (FD), air-tunnel drying (ATD) and solar drying (SD).

Fucus vesiculosus			*Gracilaria* sp.			*Ulva* sp.		
FD	ATD	SD	FD	ATD	SD	FD	ATD	SD
4.25 ± 0.71^{c}	11.51 ± 0.24^{b}	14.11 ± 0.33^{a}	3.14 ± 0.29^{b}	8.63 ± 0.24^{a}	9.13 ± 0.27^{a}	2.67 ± 0.18^{c}	11.13 ± 0.24^{b}	13.60 ± 0.40^{a}

Values are presented as mean ± standard deviation ($n = 3$). Different letters indicate statistically significant differences between drying methods ($p < 0.05$).

Differences were considered statistically significant at $p < 0.05$.

3. Results and discussion

3.1. Moisture content of the macroalgae biomass after different drying methods

The moisture content of macroalgae biomass was determined after freeze-drying (FD), air-tunnel drying (ATD), and solar drying (SD) (Table 1). Moisture content differed significantly among drying methods for all species, with FD resulting in the lowest residual moisture content (2.67–4.25 g 100 g^{-1}), whereas ATD and SD yielded higher moisture levels (8.63–14.11 g 100 g^{-1}), showing only minor species-dependent differences. Considering the significant differences in moisture content, all subsequent results for lipid content, FA, pigments, and sugar profiles were expressed on a dry weight (DW) basis, with moisture correction factor (MCF) applied to the results expressed as weight of each biomass submitted to different drying methods (Table S1).

In this study, the moisture content of *F. vesiculosus* and *Ulva* sp. under SD slightly exceeded the 12% reference threshold commonly cited in food safety standards (e.g., EU Regulation 744/2012; European Commission, 2012) and targeted for moisture-stabilized macroalgae biomass (Healy et al., 2023). However, this did not compromise the suitability of the biomass for comparative characterization, as all analyses were performed immediately after drying. As long-term storage stability was not assessed, no conclusions can be drawn regarding the stability of the dried biomass over extended storage periods. For industrial-scale applications, optimization of SD parameters may also be necessary to ensure consistent compliance with regulatory benchmarks and long-term stability for food-grade commercialization.

3.2. Effect of the drying methods on the lipid content

The total lipid content of the three macroalgal species was evaluated across different drying methods (Table 2). While no statistically significant differences were observed among the lipid content between FD, ATD, and SD in *F. vesiculosus* ($p \geq 0.05$), the lipid content in *Gracilaria* sp. significantly decreased under SD conditions (−14% relative to FD). In the case of *Ulva* sp., a significant decrease in lipid content was observed under both ATD and SD conditions (−27% and −40% relative to FD, respectively). Moreover, SD resulted in a significantly lower lipid content than ATD (−17%).

Considering the results obtained for the three macroalgae evaluated in this study, the effect of drying methods on total lipid content varied among species. In *F. vesiculosus*, the preservation of the lipid content after ATD or SD at levels comparable to the reference method (FD) suggests high stability of total lipids under the drying conditions applied. The lipid contents obtained for the three methods (4.62–5.19% DW) are in similar ranges of the values reported in the literature for this species under similar conditions, including both freeze-dried biomass (3.5–8.4% DW) (Tibbetts et al., 2016; Wirenfeldt et al., 2024) and biomass dried with hot air at 40 °C and 52 °C (2.1 and 4.1% DW) (Dhakal et al., 2024; Wirenfeldt et al., 2024). In line with our findings, Badmus et al. (2019) reported that oven-drying at 40 °C for 48 h did not negatively affect lipid content in *Fucus* spp.

In contrast, *Gracilaria* sp. showed a reduction in lipid content under SD compared to FD. The lipid contents obtained in this study for the three drying methods (2.46–2.86% DW) are within the range previously reported data for *Gracilaria* spp., including freeze-dried biomass (1.1–4.5% DW) (Francavilla et al., 2013; Khandwal et al., 2025) and oven-dried samples (1.4–3.6% DW; 25–60 °C) (Benjama and Masniyom, 2012; Freitas et al., 2021). Overall, our results suggest that, although *Gracilaria* sp. lipids can be partially preserved under milder convective drying conditions used in ATD, the exposure to moderate temperatures, such as those applied during SD, may promote lipid degradation.

The strongest effect of drying was observed in *Ulva* sp., where lipid content decreased progressively from FD to ATD and further to SD. Although the values obtained in the present study (4.09–6.78% DW) were slightly higher than the values previously reported for *Ulva* sp. biomass subjected to both FD and convective drying conditions comparable to ATD (1.5–3.4% DW) (Uribe et al., 2019; Moreira et al., 2020; Wirenfeldt et al., 2024), a clear reduction trend in the different methods was evident. In contrast, previous studies using higher drying temperatures than those applied here, such as convective airflow at 52–70 °C or sun-drying at approximately 50 °C, reported no significant differences in lipid content compared to freeze-dried *Ulva* spp. biomass (Uribe et al., 2019; Wirenfeldt et al., 2024). Variations in lipid content among studies likely reflect differences in biomass composition related to geographical origin, seasonal conditions, and cultivation factors (Moreira et al., 2021; Lafeuille et al., 2023; Djoundi et al., 2025). Moreover, the different methods employed for lipid extraction, *e.g.* adapted Bligh and Dyer method with dichloromethane/methanol applied in this study and Soxhlet-type extraction with ether used by Uribe et al. (2019), may also explain the inconsistent trends observed in the sensitivity of *Ulva* sp. lipids to drying in the different studies.

For the three species analyzed in this study, ATD and SD induced species-specific lipid losses compared to FD, which were most pronounced in *Ulva* sp. Mechanistic studies addressing the effects of drying methods on macroalgal lipid preservation remain scarce. Nevertheless, the observed species-dependent behavior suggests that intrinsic differences in the membrane/cell wall structure and composition among macroalgae may influence lipid stability during drying. In particular, the higher stability observed in *F. vesiculosus* may be associated with the thick and structurally complex cell wall typical of brown algae. The alginate-rich cell walls of brown macroalgae form a rigid ion-crosslinked matrix (Ghelichi and Jacobsen, 2025), which may reduce lipid exposure

Table 2
Lipid content (expressed as g 100 g^{-1} DW) of the biomasses of *Fucus vesiculosus, Gracilaria* sp. and *Ulva* sp. subjected to freeze-drying (FD), air-tunnel drying (ATD) and solar drying (SD).

Fucus vesiculosus			*Gracilaria* sp.			*Ulva* sp.		
FD	ATD	SD	FD	ATD	SD	FD	ATD	SD
5.19 ± 0.42	5.12 ± 0.13	4.62 ± 0.12	2.86 ± 0.13^{a}	2.66 ± 0.01^{ab}	2.46 ± 0.06^{b}	6.78 ± 0.18^{a}	4.95 ± 0.12^{b}	4.09 ± 0.19^{c}

Values are presented as mean ± standard deviation ($n = 3$). Different letters indicate statistically significant differences between drying methods ($p < 0.05$).

to oxygen and thermal stress during drying. However, this interpretation remains a hypothesis and requires further validation.

3.3. Effect of the drying methods on the fatty acid profile

Fatty acid (FA) composition was determined using two complementary derivatization approaches combined with GC–MS analysis to investigate the impact of drying on lipid stability. Alkaline-catalyzed methylation using KOH was applied to determine the profile of esterified FA within the total lipid fraction. This base-catalyzed methylation method selectively derivatizes esterified FA, while free FA remain unreacted. Acid-catalyzed using acetyl chloride allowed to analyze the total FA profile, including both free and esterified FA. A decrease in esterified FA may indicate the release of FA by lipase activity and their degradation through oxidative processes. In contrast, reductions in FA detected after acidic methylation mainly reflect oxidative degradation of FA.

For *F. vesiculosus*, a total of 15 FA were identified, with total FA (esterified and free FA) accounting for 19.03–20.66 mg g^{-1} DW (Table S2) and esterified FA accounting for 16.42–19.03 mg g^{-1} DW (Table S3). Palmitic acid (FA 16:0), oleic acid (FA 18:1 *n*−9), and arachidonic acid (FA 20:4 *n*−6) were the predominant FA (Fig. 1), consistent with previous reports for this species (Schmid et al., 2013; Maehre et al., 2014; da Costa et al., 2019; Marques et al., 2024).

The profiles of both esterified and total FA were not affected by the drying method, suggesting a high stability of nutritionally important FA across FD, ATD and SD, particularly PUFA, such as FA 18:2 *n*−6, FA 18:3 *n*−3, and FA 20:5 *n*−3. Free FA (estimated by difference between total FA and esterified FA) were found in low amounts, with no marked variation among drying methods. The most abundant free FA were saturated fatty acid (SFA) species, in particular FA 14:0 and FA 16:0 (Table S4).

Comparable FA-related nutritional values were determined for *F. vesiculosus* among the different drying methods, with no significant differences in health-related indicators (Table S3), such as the ratio of total PUFA *n*−6 to PUFA *n*−3 (*n*−6/*n*−3), and AI and TI indices. These findings are consistent with previous studies reporting no significant differences in FA profiles of *F. vesiculosus* subjected to hot air-drying at 40 °C and FD conditions (Dhakal et al., 2024). Nevertheless, previous studies on *Fucus* spp. with prolonged drying times (48 h) at 40 °C or higher drying temperatures (80 °C), conditions more comparable to the SD used in this work (22 h, 40 °C), also showed a reduction in PUFA (Badmus et al., 2019) and monounsaturated fatty acids (MUFA) (Dhakal et al., 2024), respectively.

A total of 11 FA were identified in *Gracilaria* sp., with total FA (esterified and free) accounting for 9.66–12.55 mg g^{-1} DW (Table S5) and esterified FA accounting for a total of 7.52–11.04 mg g^{-1} DW (Table S6). Palmitic acid (FA 16:0) and arachidonic acid (FA 20:4 *n*−6) were the predominant FA (Fig. 2), in accordance with literature for the same genus (Francavilla et al., 2013; da Costa et al., 2017). However, under ATD and SD conditions, the total amounts of esterified FA were reduced by 29% and 32% compared with FD, respectively. This reduction was driven by a significant decline in the content of the majority of esterified FA, except for FA 14:0, FA 15:0, and FA 18:0. Within the total FA profile (Table S5), evidence of FA oxidation was observed only for FA 18:1 *n*−7 and FA 20:4 *n*−6. Both ATD and SD reduced by 21% the absolute abundance of total FA 18:1 *n*−7, whereas the absolute abundance of total FA 20:4 *n*−6 was reduced by 27% under ATD and 28% under SD, relative to FD condition. FA 18:1 *n*−7 covers only 1% of the total FA (0.14 ± 0.01 mg g^{-1} DW in FD samples) (Table S5), therefore losses in its composition led to only a very minor change in the overall FA profile, resulting in no significant nutritional impact. The FA 20:4 *n*−6 represents the major FA in *Gracilaria* sp. biomass (6.85 ± 0.56 mg g^{-1} DW in FD samples), covering 55% of the total FA composition. Thus, significant variations in its content during drying can have a substantial effect on the overall FA profile and influence the nutritional value of the final product. Despite this reduction, no significant differences were observed in lipid quality indices, including AI and TI, among different biomasses (Table S6). Similarly to *F. vesiculosus*, free FA were estimated as the difference between total FA and esterified FA, with no statistically significant differences observed among drying methods (Table S7). Most abundant free FA were FA 16:0, FA 18:0 and FA 20:4 *n*−6. The total amount of free FA only represents 13.6%, 23.9% and 28.3% compared to those found of esterified FA in FD, ATD and SD, respectively.

Although direct evidence on the impact of drying methods on FA composition of *Gracilaria* spp. is scarce, similar reductions in total PUFA abundance under oven-drying conditions (40–60 °C) relative to FD have been reported for other red macroalgae, such as *Asparagopsis taxiformis* and *Chondrus crispus* (Regal et al., 2020; Lopez-Hortas et al., 2022).

In *Ulva* sp., 18 FA were identified, with total FA (esterified and free) accounting for 13.09–24.28 mg g^{-1} DW (Table S8) and esterified FA accounting for 6.88–20.19 mg g^{-1} DW (Table S9). Palmitic acid (FA 16:0), hexadecatetraenoic acid (FA 16:4 *n*−3), vaccenic acid (FA 18:1 *n*−7), α-linolenic acid (FA 18:3 *n*−3), and stearidonic acid (FA 18:4 *n*−3) were found as the most abundant FA (Fig. 3), in agreement with previous reports for *Ulva* spp. (McCauley et al. 2016).

Air-tunnel drying (ATD) markedly reduced the absolute amount of most esterified FA (Fig. 3b, Table S9), except for FA 18:0, FA 18:1 *n*−9

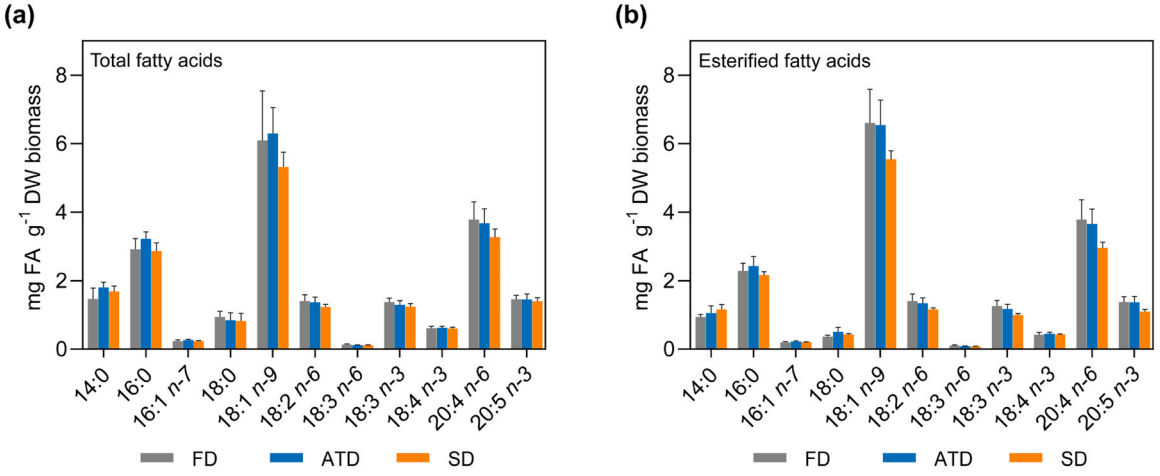

Fig. 1. Major fatty acids identified in *Fucus vesiculosus* biomass subjected to freeze-drying (FD), air-tunnel drying (ATD), and solar drying (SD). Absolute amount (expressed as mg g^{-1} DW biomass) of (a) total fatty acids obtained by acid-catalyzed methylation and (b) esterified fatty acids obtained by alkaline transesterification. Values are presented as mean ± standard deviation ($n = 3$). Detailed fatty acid profiles are shown in Supplementary Table S2 and S3.

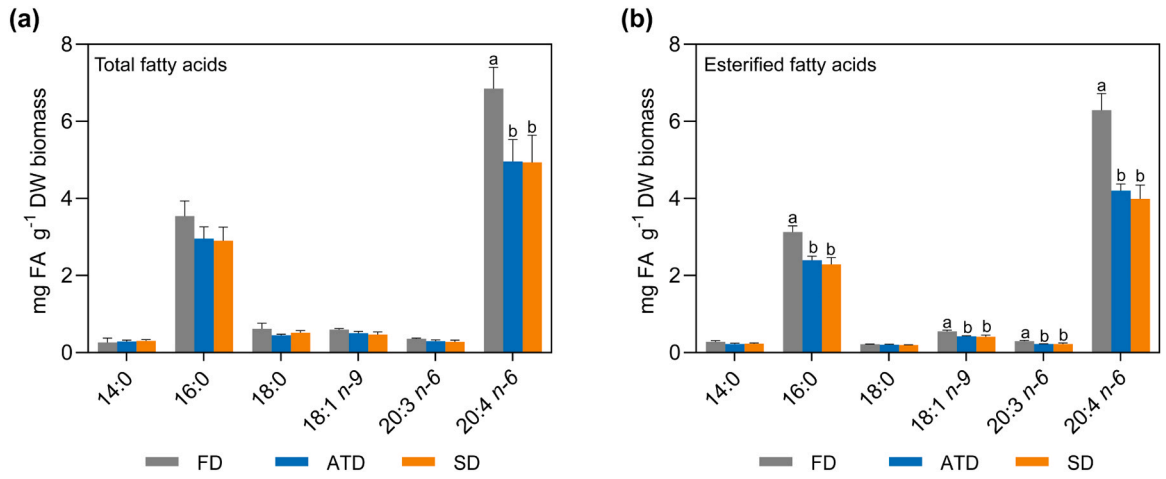

Fig. 2. Major fatty acids identified in *Gracilaria* sp. biomass subjected to freeze-drying (FD), air-tunnel drying (ATD), and solar drying (SD). Amount (expressed as mg g^{-1} DW biomass) of (a) total fatty acids obtained by acid-catalyzed methylation and (b) esterified fatty acids obtained by alkaline transesterification. Values are presented as mean ± standard deviation ($n = 3$). Different letters indicate statistically significant differences between drying methods ($p < 0.05$). Detailed fatty acid profiles are shown in Supplementary Table S5 and S6.

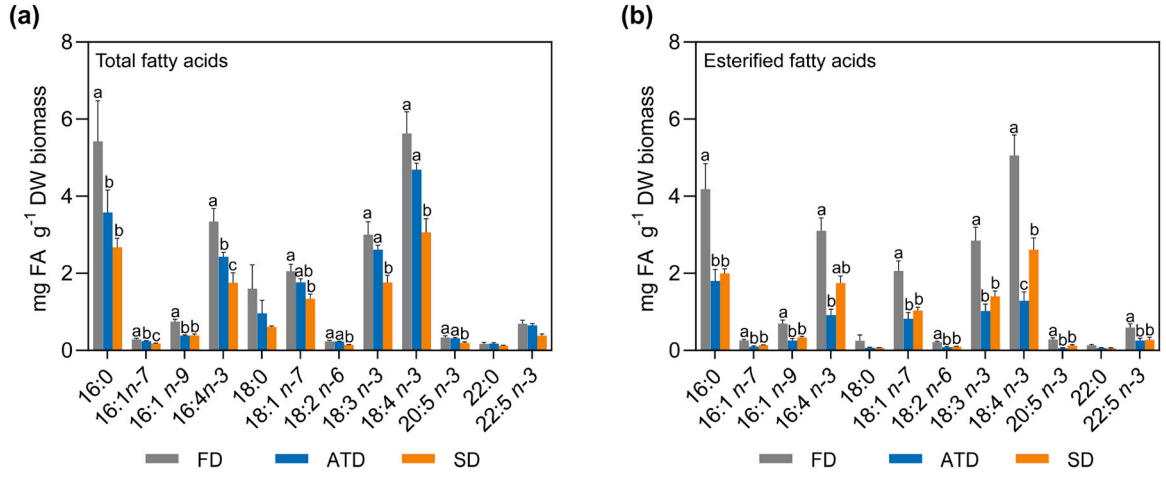

Fig. 3. Major fatty acids identified in *Ulva* sp. biomass subjected to freeze-drying (FD), air-tunnel drying (ATD), and solar drying (SD). Amount (expressed as mg g^{-1} DW biomass) of (a) total fatty acids obtained by acid-catalyzed methylation and (b) esterified fatty acids obtained by alkaline transesterification. Values are presented as mean ± standard deviation ($n = 3$). Different letters indicate statistically significant differences between drying methods ($p < 0.05$). Detailed fatty acid profiles are shown in Supplementary Table S8 and S9.

and FA 22:0. A similar, albeit less pronounced, trend was observed under SD. Consequently, the total esterified FA of *Ulva* sp. decreased by 66% under ATD and by 50% under SD compared with FD. Esterified PUFA were the most affected by ATD, showing a 70% reduction relative to FD, whereas SD led to a smaller, yet significant, decrease of 48%. Nevertheless, total FA analysis revealed a significant decrease for most FA in SD samples, except for FA 15:0, FA 18:0, FA 20:4 *n*−6, FA 22:0 and FA 22:5 *n*−3; while under ATD conditions only lower levels of 16-carbon chain FA was detected (Fig. 3a; Table S8). There was a significant accumulation of free FA (estimated by difference) in ATD biomass compared with both FD (2.9-fold increase) and SD (3.9-fold increase) conditions (Table S10). This increase was particularly evident for PUFA whose free forms increased 5.2-fold under ATD, indicating that the decrease in esterified PUFA was primarily driven by hydrolytic release rather than oxidative degradation.

In contrast, under SD conditions, the total amount of free FA in *Ulva* sp. did not differ significantly from FD. However, solar-dried biomass presented a 45% reduction in total PUFA content compared with FD biomass, suggesting a higher degree of lipid degradation under SD conditions (40 °C, 22 h). This reduction led to significant losses of health-beneficial PUFA, including linoleic acid (FA 18:2 *n*−6), α-linolenic acid (FA 18:3 *n*−3) and eicosapentaenoic acid (EPA; FA 20:5 *n*−3). Despite the reduction in total PUFA content observed under SD, the *n*−6/*n*−3 ratio remained constant regardless of the drying method (Table S8), in agreement with previous reports for *Ulva* spp. subjected to FD, vacuum drying (70 °C), convective drying (70 °C), and sun drying (50 °C with solar radiation) (Uribe et al., 2019).

Overall, our results indicate that the lipid matrix in *Ulva* sp. becomes increasingly vulnerable to thermal degradation as drying temperature rises. Drying at a moderate temperature (25 °C) still leads to some lipid degradation, but the loss of esterified FA appears to be driven mainly by the hydrolysis of structural lipids. This results in a greater preservation of FA, especially PUFA, thereby contributing to maintain the nutritional quality of the resulting dried biomass. Despite the FA losses observed under SD, health-related lipid quality indices remained largely comparable across all dried samples (Table S8).

According to the literature, lipid degradation during drying processes may also occur through FA oxidation, a process in which reactive oxygen species (ROS) preferentially target FA containing double bonds, especially PUFA species (Wang et al., 2023). This oxidative process

involves free-radical chain reactions that lead to the formation of lipid hydroperoxides as primary oxidation products. As both oxygen availability and temperature can increase FA oxidation, drying conditions involving prolonged exposure to air and moderate temperature, such as those applied in ATD and particularly in SD, may promote FA degradation by oxidation. Thus, having in consideration this interpretation, a higher susceptibility of PUFA to drying-induced degradation was observed in SD of *Gracilaria* sp. and *Ulva* sp., possibly involving oxidative processes. However, as lipid oxidation products were not directly quantified in this study, this mechanistic explanation should be considered an interpretation requiring further validation. In contrast, no clear evidence of FA degradation was detected in *F. vesiculosus* under the drying conditions tested. This species-dependent response may be related to different antioxidant capacity among macroalgae. In line, previous studies have shown that *F. vesiculosus* exhibits a higher free radical scavenging capacity compared with *Gracilaria* spp. and *Ulva rigida*, as well as other red and green seaweeds (Jiménez-Escrig et al., 2001; Silva et al., 2019). An increased radical scavenging capacity enhances protection against lipid oxidation by neutralizing free radicals, inhibiting the initiation of lipid radical formation, or quenching peroxide radicals, thereby terminating the oxidation chain reaction (Lü et al., 2010; Alvarez-Suarez et al., 2012).

Collectively, our observation emphasizes that the effect of drying conditions on FA profile is species-specific rather than determined solely by the drying procedure. Nevertheless, despite the lipid losses observed under certain drying conditions, key nutritional indicators such as *n*−6/*n*−3 PUFA ratio and AI and TI indices were maintained across drying treatments for all species.

3.4. Total chlorophylls and carotenoids content

Total chlorophylls and carotenoids contents varied markedly among species and drying methods (Fig. 4). In *F. vesiculosus*, pigment content was not significantly affected by the drying method with comparable chlorophyll and carotenoid content across FD, ATD, and SD. This stability mirrors the limited impact of drying previously observed for lipid content and FA profiles in this species. In contrast, pigment content in *Gracilaria* sp. and *Ulva* sp. was significantly influenced by the drying method. In *Gracilaria* sp., chlorophyll content decreased by 40% (from 5.90 to 3.54 mg g^{-1} DW) and by 36% (from 5.90 to 3.77 mg g^{-1} DW) under ATD and SD conditions, respectively. Nevertheless, carotenoid content declined only under ATD conditions (−39%; 0.57 mg g^{-1} DW), compared with FD (0.93 mg g^{-1} DW). In *Ulva* sp., pigment loss was even more pronounced, with ATD and SD biomass showing 56% (from 29.93 to 13.29 mg g^{-1} DW) and 64% (from 29.93 to 10.89 mg g^{-1} DW) reductions in chlorophyll content, respectively, highlighting the high susceptibility of chlorophylls in this species to drying-induced degradation. Carotenoids exhibited a similar trend, with SD causing a substantial decrease of 70% (from 7.72 to 2.27 mg g^{-1} DW), whereas ATD resulted in a non-significant decreasing trend.

As reported in the literature (Cikoš et al., 2022), pigment composition varies not only in content but also in profile among brown, red and green macroalgae, resulting in their characteristic colors. Chlorophyll *a* is a ubiquitous photosynthetic pigment in all macroalgae. Carotenoid composition varies among phyla, with fucoxanthin and violaxanthin predominating in *F. vesiculosus*, zeaxanthin and β-carotene in *Gracilaria* sp., and lutein together with β-carotene in *Ulva* sp. (Resende et al., 2023; Mutavski et al., 2024; Zepeda et al., 2024). Although individual pigment profiles were not determined in this study, and carotenoid estimation may have been influenced by chlorophyll interference inherent to spectrophotometric measurements (Pflanz and Zude, 2008; Thrane et al., 2015), the estimation of total content of chlorophylls and carotenoids using spectrophotometric approach offers valuable insights into pigment variations among biomass subjected to the different drying methods. Overall, variations in pigment content align with those observed for lipid content and FA profiles across the tested macroalgae. This trend is consistent with the lipophilic nature of chlorophylls and carotenoids, which are co-extracted with lipids. As pigments are highly sensitive to thermal degradation, freeze-dried biomass of several macroalgae generally present higher pigment content than those of hot air-dried at 40–70°C (Uribe et al., 2019; Amorim et al., 2020; Uribe et al., 2020; Nunes et al., 2024; Cuario Templonuevo et al., 2025). Nevertheless, Silva et al. (2019) reported that oven-drying at 25 °C preserved pigments in both *U. rigida* and *Gracilaria* sp. biomass, while pigment stability in *F. vesiculosus* was maintained at temperatures up to 40 °C, which is consistent with the preservation observed in this study and may contribute to enhanced protection against oxidative degradation during drying. Future studies using HPLC-based pigment profiling would enable a more detailed assessment of drying-induced changes in pigment composition.

3.5. Carbohydrate content and profile

The impact of the different drying methods on the carbohydrate content and profile was assessed in the three macroalgae (Fig. 5; Table S11). The sugars composition of *F. vesiculosus* agrees with the well-known specific polysaccharides of this species. Fucose (8.67–9.84 g 100 g^{-1} DW) was the predominant neutral sugar residue, accompanied by minor amounts of galactose (1.45–1.76 g 100 g^{-1} DW), and xylose (1.00–1.07 g 100 g^{-1} DW) residues, as well as sulphate esters (5.20–6.85 g 100 g^{-1} DW), which are characteristic constituents of fucoidans. Uronic acids (11.80–14.41 g 100 g^{-1} DW) indicates the presence of alginate, a linear polysaccharide composed of β−1,4-D-mannuronic acid and α−1,4-L-guluronic acid residues (Lee & Mooney, 2012). Glucose residues (5.02–5.39 g 100 g^{-1} DW) were also present, reflecting the occurrence of laminaran, a storage β-glucan. Mannitol was also quantified (8.12–8.67 g 100 g^{-1} DW), as it is a major photosynthetic storage compound characteristic of brown algae, and functions as both an osmoprotectant and an antioxidant (Groisillier et al., 2014). Although the drying methods did not significantly impact the total amount of carbohydrates in the three types of dried *F. vesiculosus* biomass (42.83–46.49 g 100 g^{-1} DW) (Table S11), the polysaccharides

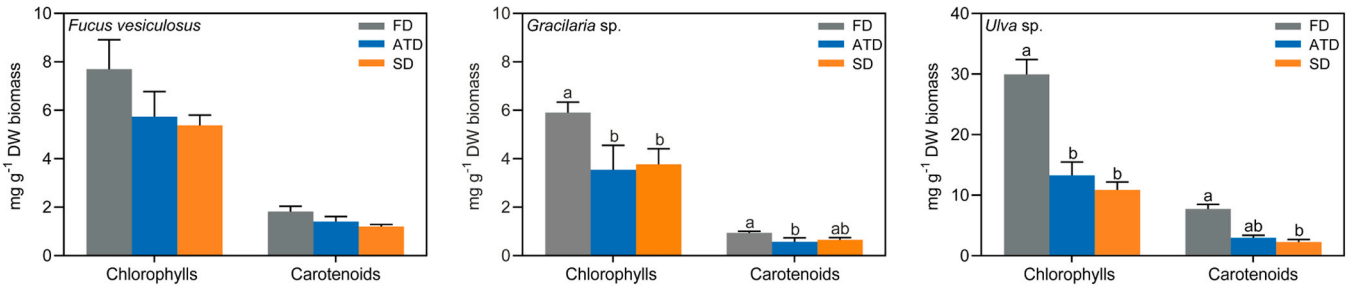

Fig. 4. Total chlorophylls and carotenoids content in biomass of *F. vesiculosus*, *Gracilaria* sp. and *Ulva* sp. subjected to freeze-drying (FD), air-tunnel drying (ATD) and solar drying (SD). Values are presented as mean ± standard deviation ($n = 3$) and are expressed as mg g^{-1} DW. Different letters indicate statistically significant differences between drying methods ($p < 0.05$).

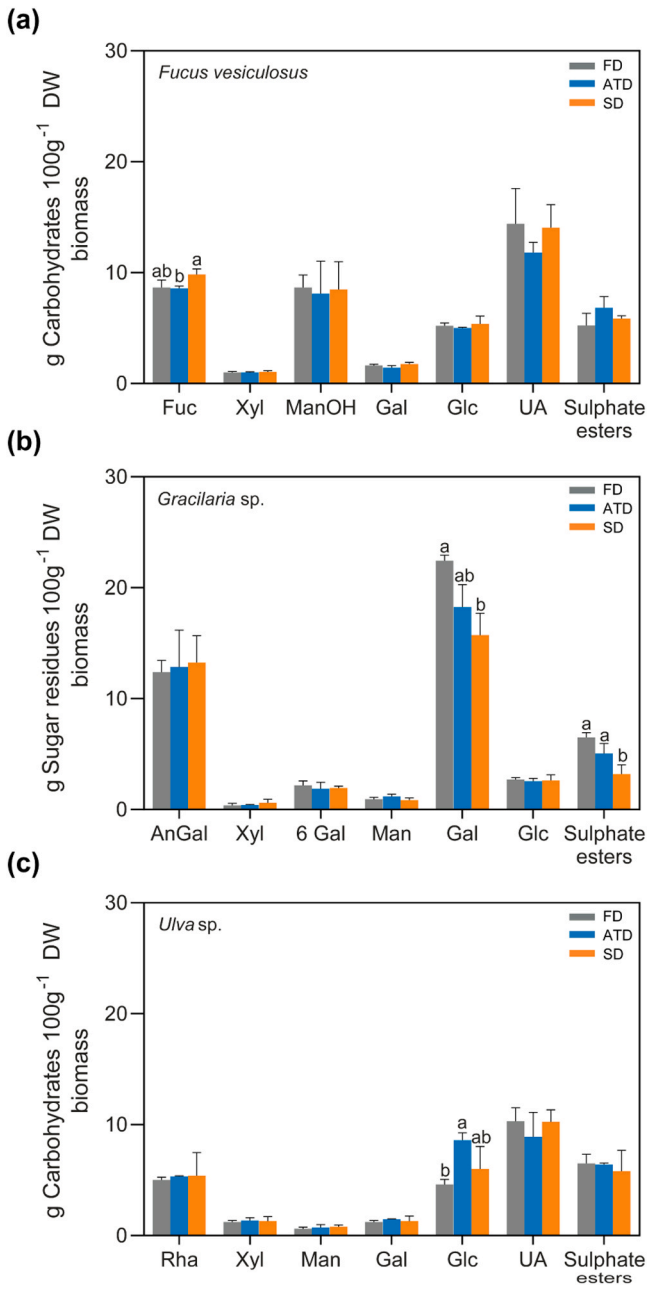

Fig. 5. Carbohydrate content in biomass of (a) *Fucus vesiculosus*, (b) *Gracilaria* sp., and (c) *Ulva* sp. subjected to freeze-drying (FD), air-tunnel drying (ATD) and solar drying (SD). Values are presented as mean ± standard deviation ($n = 3$) and are expressed as g 100 g^{-1} DW. Different letters indicate statistically significant differences between drying methods ($p < 0.05$). Rha: Rhamnose; Fuc: Fucose; AnGal: 3,6-Anhydrogalactose; Xyl: Xylose; 6 Gal: 6-*O*-Methylgalactose; Man: Mannose; ManOH: Mannitol; Gal: Galactose; Glc: Glucose; UA: Uronic Acids. Detailed monosaccharides profiles are shown in Supplementary Table S11.

profile showed some significant differences (Fig. 5a). The SD biomass had 15% higher content of fucose residues than ATD. It is known that fucoidans provide mechanical support to cell walls and protection against desiccation during air exposure of the macroalgae at low tide (Hernández-Garibay et al., 2019; Olsthoorn et al., 2021). Thus, the osmotic stress can increase crude fucoidan content to reinforce the cell wall matrix, maintain its structural integrity, and to increase hydration capacity, since fucoidans are highly hydrophilic (Bruhn et al., 2017). Based on this, it can be hypothesized that the increase in fucoidan content results from the greater dehydration stress experienced by *F. vesiculosus* during SD, owing to its prolonged exposure to a constant temperature of 40 °C, which may promote fucoidan biosynthesis compared with ATD (25 °C). Indeed, temperature can also affect fucoidan content in brown seaweeds, as previous studies have shown that higher temperatures are positively correlated with increased fucoidan levels (Ehrig & Alban, 2014; Bruhn et al., 2017). Fucoidan has antioxidant activity and, therefore, the higher fucoidan contents at higher temperatures observed during the drying process may also contribute to the protective role in eliminating ROS induced by thermal stress (Anisha et al., 2022). The impact of the drying methods on fucoidans was similar between ATD and FD. Silva et al. (2019) reported higher fucoidan extractability from *F. vesiculosus* biomass oven-dried at 25 ºC compared with FD, however, variables like drying time and fucoidans solubility should be also considered, as they may influence the final extraction yield. Although higher fucose levels were observed in SD samples, sulphate ester content did not differ significantly among the drying methods. This may indicate that higher drying temperatures induced slight desulphation of the fucoidans. Therefore, SD processing may compromise the therapeutic potential of *F. vesiculosus* fucoidans by decreasing sulphate groups, as the sulphation degree often determines their biological activities (Oliveira et al., 2017; Ferreira et al., 2021).

The carbohydrate profile of *Gracilaria* sp. was mainly composed of galactose (15.74–22.45 g 100 g^{-1} DW), 3,6-anhydrogalactose (12.39–13.26 g 100 g^{-1} DW), and 6-*O*-methylgalactose (1.90–2.19 g 100 g^{-1} DW) residues (Fig. 5b), consistent with the presence of agar, the major structural polysaccharide of this red alga. Agar is composed of agarose, a neutral polysaccharide with repeated D-galactose and 3,6-anhydro-L-galactose units linked by β−1,3- and α−1,4-glycosidic bonds, and agaropectin, which is structurally similar to agarose but contains fewer 3,6-anhydro-L-galactose units, with additional sulphate esters and other residues, such as methoxyl groups and pyruvic acid (Lee et al., 2017). The quantification of glucose residues (2.56–2.70 g 100 g^{-1} DW) suggests the presence of minor amounts of floridean starch (Rodriguez et al., 2009). The total amount of carbohydrates of the dried biomass (38.29–47.54 g 100 g^{-1} DW) (Table S11) was unaffected by the drying method used. However, the carbohydrates profile was influenced by the drying conditions, as SD led to a 30% decrease in galactose residues and a 51% reduction in carbohydrate sulphate esters when compared with the FD method (Fig. 5b). ATD conditions appear to have minimal impact on agar structure of *Gracilaria* sp., since its sugar composition was similar to that of FD biomass. These results suggest that the conditions used in SD, i.e., higher drying temperatures than FD and ATD and prolonged heat exposure (22 h) lowered agaropectin content, since both galactose residues and sulphate esters decreased. According to Macler (1988) osmotic stress can cause a decrease in agar content in the red macroalga *Gelidium coulteri* due to the decrease of photosynthesis and carbon fixation, which could possibly explain the decrease in agaropectin fraction in this study. Despite the lower agar content in *Gracilaria* sp. dried with SD, the agarose:agaropectin ratio increased, meaning that this drying method promotes an agar with higher abilities to form a firm gel. Agarose is the gelling fraction of the agar, due to the presence of 3,6-anhydro- L -galactose residues, which are responsible to constrain the molecule to form a helix, and the interactions between the helices results in gel formation (Lee et al., 2017).

Regardless of the drying method used, the carbohydrates of *Ulva* sp. biomass were mainly constituted by uronic acids (8.90–10.31 g 100 g^{-1} DW), rhamnose (5.00–5.40 g 100 g^{-1} DW), and sulphate esters (5.81–6.52 g 100 g^{-1} DW) (Fig. 5c), the main components of ulvans, the most representative polysaccharide in this species. Minor amounts of glucose, xylose, and galactose residues were also determined, which are also often reported as part of ulvan polysaccharides (Kidgell et al., 2019). Most of the glucose residues (4.61–8.61 g 100 g^{-1} DW) are possibly part of cellulose and/or starch, also characteristic of *Ulva* spp. (Prabhu et al., 2019). As observed for the other macroalgae species, although the drying method did not affect the total carbohydrates

content in *Ulva* sp. (29.54–32.83 g 100 g^{-1} DW) (Table S11), the polysaccharides were affected differently depending on the method used. While ulvan content remained similar across FD, ATD, and SD biomass, glucose residues increased by 87% in *Ulva* sp. subjected to ATD compared with FD (from 4.61 to 8.61 g 100 g^{-1} DW) (Fig. 5). During the early drying stages, *Ulva* sp. may retain some metabolic activity, although growth is inhibited. Consequently, carbon may be redirected toward polysaccharide biosynthesis, temporarily promoting its accumulation (Ribeiro et al., 2022). Under stress induced by nutrient limitation, some algae such as *Ulva ohnoi* or *Tetraselmis subcordiformis* showed a rise in starch accumulation (Prabhu et al., 2019; Pan et al., 2022). The increase of polysaccharides during abiotic (salinity) and biotic stresses was also observed in the leaves of grapevines (Oliveira et al., 2009) and olives (Cardoso et al., 2010). The lower glucose residues content observed in freeze-dried *Ulva* sp. biomass may result from the rupture of chloroplast membranes, where starch is stored, caused by ice crystal formation, which increases the exposure of starch granules to amylases and consequent metabolization (Robic et al., 2008; Zhang et al., 2014; Prabhu et al., 2019). Under ATD conditions, starch granules are potentially less exposed to enzymatic depolymerization, thereby resulting in higher measured starch content. In contrast, ulvans are cell-wall-associated sulphated polysaccharides that are less susceptible to enzymatic or physical degradation (Robic et al., 2008), explaining their apparent stability across drying treatments. In line with this, previous studies on *U. rigida* have similarly reported that ulvan recovery remains unaffected across drying methods, namely, FD and oven-drying at 25, 40, and 60 °C (Silva et al., 2019).

Overall, ATD emerged as an appropriate drying method for *F. vesiculosus*, *Gracilaria* sp., and *Ulva* sp., as it preserved the content of their specific polysaccharides, at levels comparable to those obtained with the reference method (FD) (Table S11). The preservation of the native polysaccharide structure during drying is critical for ensuring their efficacy in biological applications (e.g., antioxidant, anticoagulant, anti-inflammatory activities). Thus, the low temperature used in ATD (< 25 °C) represents a suitable industrial compromise between quality and cost for the exploitation of fucoidans, agar, and ulvans. Solar drying treatment was suitable for the stabilization of *Ulva* sp., while it may alter polysaccharides content of *F. vesiculosus* and *Gracilaria* sp., potentially affecting their characteristics; thus, their feasibility for high-value biological applications needs to be tightly controlled. Nevertheless, despite some variations in monosaccharide composition, macroalgal polysaccharides exhibited greater stability and lower susceptibility to oxidative degradation across the applied drying methods than lipids and pigments.

The long-term stability of the dried biomass was not assessed in the present study, although this is an important consideration when evaluating the overall preservation efficiency of a drying process. Previous studies have shown that storage conditions, particularly temperature and light exposure, can significantly influence the quality of dried *Ulva* and *Fucus* spp. biomass during storage periods of up to one year, resulting in PUFA losses, pigment degradation, and reductions in polysaccharide content, including alginate and fucoidan (Obluchinskaya and Daurtseva, 2020; Harrysson et al., 2021). Nevertheless, Pinheiro et al. (2019) reported no significant changes in the FA composition of dried *U. rigida* biomass after 180 days of storage, highlighting that storage stability may depend on species, processing and storage conditions. Therefore, further studies are needed to evaluate the long-term stability of dried *F. vesiculosus*, *Gracilaria* sp., and *Ulva* sp. produced using different drying methods and maintained under different storage conditions to establish product shelf life, identify optimal storage parameters, and ensure the quality of dried macroalgal biomass.

4. Conclusion

This study demonstrates that the impact of drying methods on macroalgae is strongly species-dependent. The alternative drying methods evaluated, namely ATD and SD, affected lipid and carbohydrate fractions to different extents, highlighting species-specific sensitivities to dehydration processes. Among the three species studied, *Fucus vesiculosus* exhibited high resistance to drying-induced degradation, remaining comparatively stable in all the drying conditions tested. In contrast, *Gracilaria* sp. showed moderate sensitivity to drying, particularly under SD conditions, which resulted in decreases in lipid content (−14%), esterified PUFA (−36%), chlorophyll levels (−36%), galactose (−30%), and sulphate esters content (−51%). Air-tunnel drying maintained both lipid content and carbohydrate profiles comparable to those obtained by FD, demonstrating its suitability as a milder and more cost-effective alternative method. Despite SD-induced changes, total carbohydrate content and key nutritional indices remained within comparable ranges, indicating that the overall nutritional quality of this species was largely preserved. *Ulva* sp. was the most susceptible species to drying-induced alterations, particularly within its lipid fraction, with lipid content decreasing under both ATD (−26%) and SD (−40%). Air-tunnel drying promoted hydrolytic release of FA, whereas SD led to oxidative losses of PUFA (−45%) and a reduction in carotenoid content (−70%). Nevertheless, health-related lipid indices were preserved across drying methods, suggesting that the fundamental nutritional properties of the lipid fraction were not critically compromised. Ulvan polysaccharides exhibited high stability regardless of the drying method, while starch content appeared to increase under ATD conditions. Overall, these findings emphasize the importance of selecting drying strategies that account for species-specific biochemical responses and targeted quality attributes. Drying methods should therefore be chosen based on an integrated evaluation of biomass quality, process efficiency and operational costs to maximize the nutritional and functional value of macroalgal biomass while ensuring industrial feasibility for food and bioprocessing applications.

CRediT authorship contribution statement

Mariana Neves: Writing – review & editing, Writing – original draft, Visualization, Investigation, Formal analysis. **Ana S. P. Moreira:** Writing – review & editing, Writing – original draft, Validation, Supervision. **Tiago Conde:** Writing – review & editing, Validation, Formal analysis, Data curation. **Diana Lopes:** Writing – review & editing, Writing – original draft, Validation, Conceptualization. **Beatriz Metzner:** Writing – review & editing, Visualization, Investigation. **Inês Martins:** Writing – review & editing, Visualization, Formal analysis. **Kayane Oliveira:** Writing – review & editing, Visualization, Methodology. **Andreia S. Ferreira:** Writing – review & editing, Writing – original draft, Data curation. **Cláudia Nunes:** Writing – review & editing, Validation, Supervision. **Manuel A. Coimbra:** Writing – review & editing, Visualization, Validation. **Maria Helena Cardoso:** Writing – review & editing, Validation, Methodology. **Ana Ramos:** Writing – review & editing, Methodology, Conceptualization. **Hugo Pereira:** Writing – review & editing, Validation, Funding acquisition. **Madalena Caria Mendes:** Writing – review & editing, Visualization, Formal analysis. **Inês Oliveira:** Writing – review & editing, Visualization, Investigation. **Margarida Martins:** Writing – review & editing, Validation, Investigation. **M. Rosário Domingues:** Writing – review & editing, Writing – original draft, Funding acquisition, Data curation.

Declaration of Competing Interest

The authors declare no competing interests.

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Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.jfca.2026.109393.

Data availability

Data will be made available on request.

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