Effects of drying on the nutrient content, physico-chemical and sensory characteristics of the edible kelp Saccharina latissima
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Corresponding Author:	Pierrick Stevant Aalesund, NORWAY
Corresponding Author Secondary Information:	
Corresponding Author's Institution:	
Corresponding Author's Secondary Institution:	
First Author:	Pierrick Stévant
First Author Secondary Information:	
Order of Authors:	Pierrick Stévant
	Erlend Indergård
	Aðalheiður Ólafsdóttir
	Hélène Marfaing
	Wenche Emblem Larssen
	Joël Fleurence
	Michael Y. Roleda
	Turid Rustad
	Rasa Slizyte
	Tom Ståle Nordtvedt
Order of Authors Secondary Information:	
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	Sparebanken Møre Mr Pierrick Stévant

Abstract:	The effects of convective air-drying at 25, 40 and 70°C and freeze-drying on the quality of the edible kelp Saccharina latissima to be used for food was investigated. Based on the analysis of the carbohydrate and amino acid profiles, as well as polyphenol, fucoxanthin and ash contents, no significant differences were detected among sample groups and air-drying up to 70°C results in equally nutritious products at shorter processing times. Only the iodine content was found lower in freeze-dried compared to air-dried samples. The swelling capacity of the air-dried samples was significantly lower than in freeze-dried samples, particularly at high temperatures (40 and 70°C), reflecting alteration of the physico-chemical properties of the seaweed during air-drying (attributed to product shrinkage) and reduced capacity of the final product to rehydrate. Structural differences between air-dried products at 25 and 70°C may explain the differences in mouthfeel perception (dissolving rate) among the two sample groups observed during a sensory evaluation. Overall the drying temperature within this range did not alter the aroma (i.e. odor) nor the flavor intensity of the product. In food

	applications where the product's mechanical properties (e.g. porosity) are essential, freeze-drying, and to a lesser extent air-drying at low temperatures, will result in higher quality products than air-drying at higher temperatures.
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1 **Effects of drying on the nutrient content, physico-chemical and sensory characteristics of the edible kelp**

2 *Saccharina latissima*

3 Pierrick Stévant [a,b,1], Erlend Indergård [c], Aðalheiður Ólafsdóttir [d], Hélène Marfaing [e], Wenche Emblem Larssen [a],

4 Joël Fleurence [f], Michael Y. Roleda [g], Turid Rustad [b], Rasa Slizyte [c], Tom Ståle Nordtvedt [c]

5

6 [a] Møreforsking Ålesund AS, PO Box 5075, 6021 Ålesund, Norway

7 [b] Norwegian University of Science and Technology NTNU, 7491 Trondheim, Norway

8 [c] SINTEF Ocean, PO Box 4762, Torgard 7465 Trondheim, Norway

9 [d] Matís ohf, Vinlandsleið 12, 113 Reykjavík, Iceland
10
11 [e] CEVA (Centre d'Etude et de Valorisation des Algues), B.P. 3, F-22610 Pleubian, France

12 [f] MMS (Mer Molécule Santé), EA2160, Université de Nantes, BP 92208, 44322 Nantes, France

13 [g] Norwegian Institute of Bioeconomy Research, 8027 Bodø, Norway
14

15 **Abstract**

16 The effects of convective air-drying at 25, 40 and 70°C and freeze-drying on the quality of the edible kelp

17 *Saccharina latissima* to be used for food was investigated. Based on the analysis of the carbohydrate and amino

18 acid profiles, as well as polyphenol, fucoxanthin and ash contents, no significant differences were detected among

19 sample groups and air-drying up to 70°C results in equally nutritious products at shorter processing times. Only

20 the iodine content was found lower in freeze-dried compared to air-dried samples. The swelling capacity of the

21 air-dried samples was significantly lower than in freeze-dried samples, particularly at high temperatures (40 and

22 70°C), reflecting alteration of the physico-chemical properties of the seaweed during air-drying (attributed to

23 product shrinkage) and reduced capacity of the final product to rehydrate. Structural differences between air-dried

24 products at 25 and 70°C may explain the differences in mouthfeel perception (dissolving rate) among the two

25 sample groups observed during a sensory evaluation. Overall the drying temperature within this range did not

26 alter the aroma (i.e. odor) nor the flavor intensity of the product. In food applications where the product's

27 mechanical properties (e.g. porosity) are essential, freeze-drying, and to a lesser extent air-drying at low

28 temperatures, will result in higher quality products than air-drying at higher temperatures.

29 **Key words:** air-drying; freeze-drying; nutrients; physico-chemical properties; seaweed; sensory

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[1] Corresponding author: email: pierrick.stevant@moreforsk.no, tel: +47 70 11 16 13

Introduction

Seaweeds have been used for centuries in Asian cuisine for their nutritional properties as well as for their rich and unique flavors. In Western countries, macroalgae have not been a significant food source throughout history and industrial applications have long been limited to the extraction of phycocolloids (alginate, agar, carrageenan) for the food industry. Seaweeds belong to a diverse group of photosynthetic marine plants, with a variable chemical composition depending on species, season and habitat, and the nutritional value of several species along with their health benefits have been reviewed (Holdt and Kraan 2011; Déléris et al. 2016). Most species are characterized by high levels of dietary fibers and minerals, and low lipid levels (MacArtain et al. 2007; Dawczynski et al. 2007). Their protein composition (Fleurence 2004; Dawczynski et al. 2007; Mæhre et al. 2014) and antioxidant activities, associated to their content in polyphenolic compounds (Wang et al. 2012) and pigments (e.g. fucoxanthin) (Fung et al. 2013) make seaweed an attractive raw material for the provision of bioactive substances with a broad range of applications, especially in human and animal nutrition. In addition to their nutritional benefits, edible seaweeds, including common species along the coast of Europe, have both flavor enhancing (Mouritsen et al. 2012; Chapman et al. 2015; Mouritsen 2016) and physico-chemical properties (texture, water- and fat binding properties, color) (Cofrades et al. 2008; Chapman et al. 2015) that can be applied to the field of gastronomy and to the food industry. Hence, seaweeds can be included in a wide range of foodstuffs and are increasingly recognized as versatile and delicious whole foods, promoted by health food trends and the use of locally available natural ingredients.

In Europe where the potential of seaweeds in various industrial applications has triggered the interest to cultivate biomass, a number of commercial initiatives have emerged in recent years (Stévant et al. 2017c). Large-scale seaweed cultivation largely focuses on kelp species, especially *Saccharina latissima*, due to its phytochemical content and ability to achieve high biomass yields in short time. Moreover, this species, which is closely related to the Japanese *konbu* (*Saccharina japonica*), is prized for its flavor as well as high levels of potassium compared to sodium salts, with potential as salt replacing ingredient in the food industry resulting in healthier mineral profiles in manufactured food products (Rioux et al. 2017). On the other hand, the particularly high iodine content of *S. latissima* (Stévant et al. 2017a; Roleda et al. 2018), could have negative consequences on human health, especially in sensitive individuals, if large amounts of this seaweed are ingested regularly over an extended period (Miyai et al. 2008). However, these levels can be reduced by processing in the perspective of an extensive use in the food industry (Lüning and Mortensen 2015; Stévant et al. 2017a). Although product development from cultivated seaweeds is still limited, products with relatively high market value such as foods and food ingredients, are predicted to play an important role in creating value from farmed seaweeds (Stévant et al. 2017c).

61 Kelp species are characterized by a high moisture content and rapid microbial decomposition once harvested

62 (Enríquez et al. 1993) thus require adequate pre-treatments to maintain product quality and ensure consumer

63 safety. Although several alternatives are available (e.g. salting, freezing), drying is the preferred method for

64 stabilizing seaweed biomass for long-term storage. However, the effects of preservation treatments including

65 drying, on the quality parameters of seaweed biomass is a major question which has only partially been studied.

66 Previous studies on the pre-treatment of the brown macroalgae *Sargassum* spp. suggest that freeze-drying is the

67 most appropriate drying method providing products with higher nutrient content when compared to convective

68 air-drying methods (Chan et al. 1997; Wong and Cheung 2001a). Generally, due to the absence of liquid water

69 and to the low temperatures during the process of freeze-drying biomaterials, the rate of most reactions responsible

70 for the product deterioration are very low, resulting in high quality products (Bonazzi and Dumoulin 2011). On

71 the other hand, freeze-drying is associated with high equipment and operation costs, along with slow drying rates,

72 making this technology less attractive than conventional convective air-drying in commercial settings. The effects

73 of certain drying conditions, e.g. temperature, on specific compounds or characteristics of some seaweed species

74 are reported. Generally, higher drying temperatures lead to a reduction in phytochemical substances such as

75 phenolic compounds (Moreira et al. 2016) and pigments (Tello-Ireland et al. 2011) together with modifications

76 of the physico-chemical (Tello-Ireland et al. 2011; Sappati et al. 2017) and sensory properties (Michel et al. 1997)

77 of the seaweed products. However, systematic knowledge on the effects of drying treatments on the overall quality

78 of edible kelps of commercial importance, including *S. latissima*, is still missing.

79 The objective of this study is to characterize and compare the quality of *S. latissima* stabilized by different drying

80 methods, i.e. convective air-drying (referred to as air-drying) at different temperatures compared to freeze-drying.

81 Quality was defined as the nutrient content determined by the analysis of bioactive substances including proteins

82 and amino acids, mineral fraction, carbohydrates, polyphenol and fucoxanthin pigment in the dried products. In

83 food applications, the sensory and physico-chemical characteristics (i.e. water and fat binding properties,

84 swelling) along with the product's appearance are important factors determining consumer acceptance. Hence,

85 these parameters are included in the comparative quality assessment of *S. latissima* following different drying

86 treatments. Understanding the behavior of the seaweed biomaterial is a key to develop processing strategies that

87 will maximize the quality of the products to be used as food ingredients and as raw material for the provision of

88 valuable compounds.

89

90 **Materials and methods**

91 *Biomass harvest and drying treatment*

92 Samples of *S. latissima* were harvested from SINTEF's cultivation site, off the coast of Hitra in Norway on May

93 18 and 19, 2016. Batches of 25 kg seaweed biomass were stored in airtight and refrigerated containers during

94 transport to the laboratory where simultaneous drying experiments were conducted, i.e. air-drying at 25, 40 and

95 70°C and freeze-drying.

96 Air-drying treatments were performed in shelf dryers where 25 to 30 kg of seaweed (mature adult thalli) were

97 scattered as monolayers to avoid uneven dying of the material (case hardening). The initial stocking density was

98 approximately 1.4 kg m^2, with a shelf area of 0.4 m^2. Drying at 40 and 70°C was achieved by indirect heating of

99 the air by liquefied propane gas, while a heat pump system was used to produce drying air at 25°C. The air velocity

100 between the shelves was in the range of 1.5 to 3.0 m sec^{-1}. The temperature and relative humidity (RH) were

101 monitored during the drying process (see fig. S1, online resource 1). The samples' weight was measured at regular

102 intervals until equilibrium moisture content (EMC) was reached and no further variations were observed.

103 Simultaneously, samples were vacuum-packed and frozen for subsequent vacuum freeze-drying (Alpha 2-4 LSC).

104 All treatments were performed in three replicates. All dried samples were vacuum-packed and dispatched for

105 further analyses.

106 *Chemical Analyses*

107 *Moisture* The moisture content in the dried samples was determined gravimetrically by drying at 105^{0}C until

108 constant weight of the samples was achieved (typically 24 h). The subsequent results from chemical analyses were

109 then expressed as part of the dry weight (DW) of the samples.

110 *Ash content* was determined after combustion of the dried samples at 590^{0}C for 12 h in a laboratory muffle

111 furnace. The ashes were quantified as the residue from combustion expressed as percentage of the DW.

112 *Iodine* Dried seaweed samples were ground to 120 µm grain size using an electric grain miller and iodine (I) was

113 extracted by dry alkaline incineration, a process where all inorganic and organic iodine species were converted to

114 iodide (I^-) ions. Thereafter, the iodide in algal extracts, as a measure of the total iodine in algal samples, was

115 quantified using a HPLC system (1200 Series, Agilent Technologies, Palo Alto, USA) according to Nitschke and

116 Stengel (2015). An Acclaim Mixed-Mode WAX-1 column, protected by an Acclaim Mixed-Mode WAX-1 guard

117 column (Dionex Corporation, Sunnyvale, USA) was used to separate iodide ions from interfering compounds.

118 The mobile phase was 50/50 (v/v) methanol/phosphate buffer. The iodide eluted was detected by a diode array

119 detector at 223 nm, identified via retention time and absorption characteristics, and quantified by peak area. The

120   HPLC method used has a limit of detection (LOD) and a limit of quantification (LOQ) of ~0.2 ng μL^{-1} and 1 ng

121   μL^{-1}, respectively. Iodine contents were expressed in mg g^{-1} DW.

122   **Carbohydrate analysis** Neutral sugars (D-glucose, D-galactose, D-mannose, D-xylose, L-fucose, L-rhamnose),

123   D-mannitol and uronic acid (D-glucuronique, D-mannuronic, poly-D-guluronic and poly-D-mannuronic)

124   composition were determined by high-performance liquid chromatography (HPLC) analysis after

125   depolymerization under methanol- acid hydrolysis reaction (methanolysis) as described by Quemener et al.

126   (2000). Ground freeze-dried seaweed samples of 15 mg were transferred into 2 mL MeOH–HCl solution, prepared

127   by adding acetyl chloride in methanol (17/3 v/v, from pure solutions). Methanolysis was conducted at 100°C for

128   4 h, after which neutralization was achieved by adding silver carbonate (successively 100 mg then 50 mg) until

129   pH reached 4-5. The solutions were evaporated at 47°C for 16 h, then dissolved in distilled water and filtered prior

130   to HPLC analysis (Grace smart RP18, 5 μm, 4.6×250 mm). Chromatographic peaks were identified by comparison

131   with high purity reference sugars purchased from Sigma-Aldrich (Steinheim, Germany) except for the poly-D-

132   guluronic and poly-D-mannuronic standards prepared at the laboratory. The sum of guluronic and mannuronic

133   acids (known as G- and M-units) measured in the samples, which are the monomeric units composing alginate,

134   was used to quantify the alginate content. The laminaran content of the samples was quantified by the glucose

135   levels measured in the hydrolysates. Results were expressed as % of the DW.

136   **Total nitrogen (N)** was determined in ground samples using a CHNS-O elemental combustion system (Costech

137   Instruments ECS 4010) at a temperature of approximately 1000°C, where the N of the samples is converted to N

138   gas/oxides. The measurements were performed in 4 parallels. Results were expressed as % N of the DW.

139   **Amino acid analysis** The amino acid profiles were analyzed from ground samples by a HPLC system (Agilent

140   Infinity 1260, Agilent Technologies) coupled to an on-line post-column derivatization module (Pinnacle PCX,

141   Pickering laboratories, Mountain View, CA, USA), using ninhydrin (Trione) as a reagent and a Na^{+}-ion exchange

142   column (4.6 x 110 mm, 5 mm). 18 standard amino acids and taurin were quantified from standard curves measured

143   with amino acid standards. Prior to the analysis, the samples were hydrolyzed in 6 M HCl containing 0.4%

144   mercaptoethanol for 24 h at 110°C (HCl hydrolysis). Glutamine (Gln) and asparagine (Asn) were converted to

145   glutamic (Glu) and aspartic acid (Asp), respectively. Cystein (Cys) was quantified as cystin (Cys-Cys). The

146   samples were filtered using a micro-filter, the pH was adjusted to 2.2 and the samples were further diluted with a

147   citrate buffer (pH 2.2) for the HPLC analysis. All buffers, reagents, amino acid standards and the column were

148   obtained from Pickering laboratories (Mountain View, CA, USA). HCl and mercaptoethanol were obtained from

149   Sigma-Aldrich.

150 ***Free amino acid analysis*** The free amino acid content of the samples was determined using the method of Osnes

151 and Mohr (1985). The proteins were extracted by agitating 100 mg of ground dried sample in 10 mL water for 1

152 h. The extract was centrifuged at 4°C and 2000 g for 20 min. 0.25 ml of 10% sulphosalisylic acid was added to 1

153 mL of the water-soluble extract in an Eppendorf tube. The mixture was then vigorously shaken and incubated at

154 4°C for 30 min prior to centrifugation at 7840 g for 10 min in order to precipitate the protein-bound amino acids.

155 1 mL of the supernatant was transferred to a new Eppendorf tube with 0.25 mL of 10% sulphosalisylic acid and

156 the same operation as previously described was repeated until no protein precipitate was observed. The analysis

157 was done in triplicate. Suitably diluted samples were filtered (0.2 µm) prior to analysis by HPLC (Dionex Ultimate

158 3000) using a Water Novapak C18 column (4.0 µm particle size) and a RF 2000 fluorescence detector (Dionex,

159 Sunnyvale, CA, USA). The free amino acids were identified and quantified by comparison with pure amino acid

160 standards purchased from Fluka (Buchs, Switzerland). Both cysteine and proline were excluded from the analysis,

161 cysteine being unstable during the acid hydrolysis of the samples and proline cannot be detected following the o-

162 phtalaldehyde (OPA) pre-column derivatization during the HPLC analysis. The results were expressed in mg g^{-1}

163 DW of the seaweed samples.

164 ***Polyphenolic content*** The polyphenolic content of algal extracts was determined colorimetrically using the Folin–

165 Ciocalteu reagent according to the method of Ragan and Glombitza (1986). The extraction was performed using

166 250 mg of ground freeze-dried seaweed samples in 10 mL solvent (acetone/water, 80/20 v/v). The mixture was

167 incubated for 1 h in the dark at room temperature. After decantation, the supernatant was recovered and re-

168 extracted under the same conditions. Both supernatants were pooled prior to filtration (0.45 µm). The filtrate

169 represented the seaweed sample extract. Then, 200 µL of seaweed extract was mixed with 1300 µL distilled water

170 and 100 µL Folin-Ciocalteu reagent followed by the addition of Na_2CO_3 (29%). After incubation at 45°C for 30

171 min in the dark, the absorbance was recorded at 760 nm using a UVIKON-XL spectrophotometer (Bio-Tek

172 Instruments, USA), with phloroglucinol used as the standard reference (Sigma-Aldrich, Steinheim, Germany). A

173 standard curve with serial phloroglucinol solutions (ranging from 0 to 100 µg ml^{-1}) was used for calibration. The

174 polyphenol contents were expressed as phloroglucinol equivalent in % of the DW. Analyses were performed in

175 duplicate with 10% relative uncertainty of measure.

176 ***Fucoxanthin content*** The extraction of fucoxanthin from air-dried samples at 25 and 70°C and freeze-dried

177 samples was carried out in ethanol/water solvent (60/40) for 2 h in ice bath protected from light (1% seaweed

178 powder in solvent). After decantation, the seaweed sample residue was subjected to a second extraction following

179 the same conditions. The supernatants were pooled prior to analysis. The fucoxanthin content in the extracts was

180   determined by reversed phase HPLC in a YMC Carotenoid column (250 x 4.6 mm i.d. 5.5 µm particle size,

181   INTERCHIM, France) with UV detection at 448 nm. Acetonitrile, methanol and water was used as mobile phase.

182   A commercial fucoxanthin standard (C5753, Caroténature) was used for quantification.

183   *Color and physico-chemical properties*

184   *Surface color analysis* The surface color of seaweed samples was analyzed by a computerized image technique

185   as described by Girolami et al. (2013), using a digital camera (Canon EOS 60D) and a 35 mm lens (Canon EF 35

186   mm f/2) mounted in a black box isolated from any external light. Lighting was achieved with two fluorescent light

187   bulbs with a color temperature of 6500 K (D_{65}, standard light source commonly used in food research) positioned

188   at an angle of 45° from the sample to obtain uniform lighting. The color was analyzed quantitatively using

189   Photoshop (Photoshop CC 2017, Adobe Systems Inc.) and expressed in CIE *L** (whiteness or brightness), *a**

190   (redness/greenness) and *b** (yellowness/blueness) coordinates, as described by Yam and Papadakis (2004).

191   *Water and oil binding capacity (WBC and OBC)* was determined as described by Rupérez and Saura-Calixto

192   (2001) where 30 mL of either distilled water or a commercial soya oil was added to 0.5 g ground samples (particle

193   size 0.8 mm) in a 50-mL centrifuge tube. The samples were then stirred and left at room temperature for 1 h. After

194   centrifugation at 3000 g for 20 min, the supernatant was discarded and the residue weighed. WBC and OBC were

195   expressed as g water per g of dried sample.

196   *Swelling capacity (SC)* was assessed following the method described by Rupérez and Saura-Calixto (2001) and

197   slightly modified, where 1 to 2 g ground samples was added to a 50-mL measuring cylinder. 30 mL of distilled

198   water was added under agitation using a vortex mixer to eliminated trapped air bubbles. The samples were covered

199   and left overnight then SC was determined as the volume occupied by the sample (in mL) per g of dry sample

200   initially added. The analysis WBC, OBC and SC of each sample was performed in 3 parallels.

201   *Sensory analysis*

202   A descriptive test (ISO:13299, 2003) was used to characterize the sensory profile of *S. latissima* samples air-dried

203   at 25 and 70°C. The panel consisted of eight judges, ranging from 31 to 60 years of age, all of which had some

204   experience with descriptive analysis but were not familiar with testing seaweeds. Assessors were trained according

205   to the guidelines in ISO:8586:1 (2012).

206   The seaweed samples were pulverized using a blender and presented to the assessors in small beakers (1-2 g per

207   assessor). During a first training phase, the assessors developed a vocabulary describing the samples' odor

208   (aroma), flavor and texture characteristics, and agreed upon a total of 13 attributes listed and described in table 1.

209   Samples of *S. latissima* produced from different pre-treatments (4 in total) were used in this sensory evaluation

210 although only the results concerning the air-dried samples at 25 and 70°C are relevant to this study and will be

211 discussed. Several pretest sessions were conducted as described by Lawless and Heymann (2010), in which the

212 panel members were trained in the evaluation of the attributes by testing samples that were characteristic. A

213 continuous non-structured scale was used for the evaluation, ranging from lowest to highest intensity,

214 corresponding to the range of intensity of the tested samples with regard to each attribute. The results from

215 panelists were transformed to numbers from 0 to 100 (lowest to highest intensity) for the data analysis. The

216 evaluation followed detailed instructions in which the panel members evaluated the aroma of the samples by

217 smelling prior to evaluating their flavor and texture attributes. The training was conducted during two days before

218 the main test and resulted in a calibrated panel.

219 During the main evaluation phase, each assessor performed a monadic assessment of the seaweed samples using

220 a computerized system (surveymonkey.com) for direct recording of the data. The evaluation was performed in

221 two replicates. Panel performance was monitored using PanelCheck Software (version 1.3.2, Nofima, Tromsø,

222 Norway).

223 *Statistical analysis*

224 All statistical analyses were performed on R (version 3.4.1, R Development Core Team 2017). Raw data were

225 pre-processed for descriptive statistics and the results expressed as mean ± standard error (n = 3) unless stated

226 otherwise. A one-way analysis of variance (ANOVA, R function aov) was used to detect significant differences

227 among treatment groups regarding individual quality parameters, after testing for the homogeneity of variances

228 (Levene test). A Tukey's honest significant difference (HSD) test (R function TukeyHSD) was used for post-hoc

229 comparisons of significant ANOVA results. A principal component analysis (PCA, R function prcomp) based on

230 covariance matrix was applied to visualize differences in the amino acid and free amino acid compositions among

231 treatment groups. A PCA based on correlation matrix, in which variables of different scales are standardized, was

232 used to detect differences in color characteristics.

233

234 **Results and discussion**

235 *Experimental drying*

236 Freshly harvested biomass of *S. latissima* initially containing 89.5 ± 0.4% (n = 10) water was air-dried at 3

237 different temperatures (25, 40 and 70°C) and freeze-dried (used as reference treatment). The experimental drying

238 kinetics of air-dried samples is shown in figure 1. EMC at 25, 40 and 70°C was achieved at 420, 270 and 100 min

239 respectively. In comparison, freeze-drying of fresh *S. latissima* samples was achieved during a 20 h cycle. The

240   levels of residual moisture were significantly different among samples (ANOVA: $F_{(3, 8)}$ = 10.69, p = 0.004; table

241   2) with higher levels found in air-dried samples at 25°C, compared to other groups. This result can be explained

242   by higher RH levels measured at this temperature using the heat pump drying system compared to air-drying at

243   40 and 70°C using a classical indirect air heating system (see fig. S1, online resource 1). Increasing RH decreases

244   the drying rate due to lower mass transfer coefficient (Sappati et al. 2017). For an accurate comparison of the

245   quality of the samples obtained from different treatments, the following results from the chemical analyses were

246   adjusted to the residual moisture of the samples and expressed on a DW basis.

247

248   *Nutrient content*

249   The effects of drying treatments on the nutrient content of the raw material cultivated in Norway and harvested in

250   May, were assessed by chemical composition. Table 2 summarizes the results from the chemical composition of

251   the samples from 4 drying treatment groups, including residual moisture, ash and iodine content, carbohydrate

252   composition, polyphenols and fucoxanthin contents. The lipid content was not analyzed in this study but is

253   reported to be low in brown macroalgae in general (MacArtain et al. 2007; Dawczynski et al. 2007) and ranging

254   from 0.8 to 2% DW in *S. latissima* (Gómez-Ordóñez et al. 2010; Sappati et al. 2017).

255

256   The chemical composition of the samples is dominated by their ash content (ca. 45% DW, table 2), directly

257   reflecting the high mineral content of the samples, followed by carbohydrates (ca. 25% DW). Substantially higher

258   carbohydrate and lower ash contents in freeze-dried *S. latissima* samples also harvested in May are reported in

259   the literature (Schiener et al. 2015; Stévant et al. 2017b) highlighting the variability in the chemical composition

260   of this kelp species among geographical regions. There were no significant differences in ash content among air-

261   and freeze-dried samples. Particularly high levels of iodine in *S. latissima* are reported in the literature (Stévant et

262   al. 2017a; Roleda et al. 2018), with a potentially negative impact on its nutritional value since excessive iodine

263   intakes can be associated with clinical symptoms in sensitive individuals (Miyai et al. 2008). The iodine content

264   was significantly lower in freeze-dried compared to air-dried samples (ANOVA: $F_{(3, 7)}$ = 17.17, p = 0.002). In

265   kelp species, the iodine accumulates naturally in the extracellular matrix in the form of iodide (I^-) which readily

266   scavenges a variety of reactive oxygen species (ROS) from both aqueous and gaseous oxidants (Küpper et al.

267   2008). A study from Hou et al. (1998) reported reduced recovery of iodide in aqueous solutions following freeze-

268   drying (30 to 40%) compared to air-drying (100%) although a similar effect of freeze-drying on the recovery of

269   iodide directly from seaweed material was not observed. However, the chemical species of iodine are known to

270 differ among seaweeds and no mention is made of the species used in this study. The mean iodine content of

271 freeze-dried *S. latissima* from a large-scale sampling program (4.6 mg g^{-1} DW), including samples of the same

272 biomass as used in this study (Roleda et al. 2018), is comparable to the values for air-dried samples presented in

273 table 2. In the present study, the iodine level of freeze-dried samples is lower than in air-dried samples. However,

274 it is in the lower range of the values (ranging 1.6 – 7.2 mg g^{-1} DW) reported by Roleda et al. (2018) across spatial

275 and temporal variations of the biomass source. These results should be interpreted with caution due to the small

276 sample size and contradictory findings from Nitschke and Stengel (2016) reporting no differences in iodine

277 content between freeze-dried and air-dried samples, from the species *Alaria esculenta*, *Palmaria palmata* and

278 *Ulva intestinalis*.

279 The total carbohydrate (TC) content, which was quantified as the sum of each individual sugar identified, did not

280 significantly differ among samples (table 2). The carbohydrate fraction was mainly composed of alginate, reaching

281 over 50% of the TC, followed by mannitol (approximately 25% of TC). The methanol-acid hydrolysis reaction

282 (methanolysis) only allows for the detection of soluble sugars present in the samples. The insoluble fibers fraction,

283 mainly found in kelps species within the cell walls in form of cellulose, cannot be quantified by this method. A

284 study from Schiener et al. (2015) reports stable cellulose contents across seasons in *S. latissima* accounting for

285 11% of the DW. The fucose, mainly present in sulfated form in brown seaweeds, is indicative of the fucoidan

286 content of the samples. Both laminaran and fucose are accounting for less than 10% of the TC. Galactose, mannose

287 and glucuronic acid, which enter into the composition of fucoidans in *S. latissima* (Marfaing et al. 2009), were

288 also detected in small amounts in all samples, at levels below 1% DW. The levels of individual sugars did not

289 notably differ among sample groups suggesting no effects of the drying treatments on the carbohydrate

290 composition of *S. latissima*.

291

292 Lower polyphenol contents are reported in *Sargassum* spp. following air-drying at 60°C compared to freeze-

293 drying (Wong and Cheung 2001b) and decreasing levels of phenolic compounds as well as antioxidant activity in

294 *Fucus vesiculosus* were observed from increasing drying temperature (from 35 to 75°C, Moreira et al. 2016). In

295 contrast, Gupta et al. (2011) reported higher loss of total phenolic content in *Himanthalia elongata* following air-

296 drying at 25°C compared to 40°C, which can be explained by higher enzymatic oxidative activity in the material

297 dried at lower temperature. The polyphenol levels of dried *S. latissima* samples measured in this study were low

298 and did not significantly differ among drying treatments (table 2). Relatively lower fucoxanthin contents were

299 measured in the samples air-dried at 25°C compared to 70°C and freeze-dried although this trend was not

300 significant (ANOVA: $F_{(2, 6)} = 4.88$, $p = 0.07$) due to the variability observed within treatment groups. These

301 results are contradictory to those obtained from a similar experiment conducted on *A. esculenta* in which air-

302 drying at 70°C produced samples with the lowest fucoxanthin content (Stévant, unpublished results). Moreover,

303 the sensitivity of this carotenoid pigment to high temperatures has previously been reported (Indrawati et al. 2015).

304 On the other hand, the longer drying time at 25°C may result in increased oxidation of the pigment. Low drying

305 temperatures may also fail to inactivate oxidative enzymes responsible for pigment degradation, although further

306 work on the fucoxanthin stability following preservation treatments of brown seaweeds is needed in order to better

307 understand the behavior of this compound in the raw material.

308

309 The protein content of the samples, reflected by the sum of all amino acids analyzed, ranged from 7.2 to 7.4%

310 DW (table 3) which is comparable to values reported in the literature for *S. latissima* (Schiener et al. 2015; Stévant

311 et al. 2017b). These levels did not vary among drying treatments, in contrast with the results obtained by Chan et

312 al. (1997) and Wong and Cheung (2001a) who detected lower levels of total amino acids in *Sargassum* spp.

313 samples air-dried at 60°C compared to freeze-dried samples. In both cases the protein loss was non-specific since

314 the relative amounts of individual amino acids remained constant. The amino acid composition of the *S. latissima*

315 samples in this study was dominated by glutamic acid, alanine and aspartic acid representing approximately one

316 third of the protein fraction in all sample groups. All essential amino acids (EAA) were detected in the samples

317 except tryptophan which can be destroyed during the acid hydrolysis of the samples. The PCA method only

318 explained 65.1% of the total variation (cumulated by the two principal components) in amino acid composition

319 among samples hence, was excluded from the analysis of the results. However, overlapping values of mean ±

320 standard error for individual amino acids across sample groups (table 3) suggest no effect of the drying treatments

321 on the protein quality of *S. latissima*.

322 Although the protein content of brown seaweeds is generally lower than those found in red and green species

323 (Fleurence 2004), the interest in large-scale cultivation of kelp, primarily *S. latissima*, is growing rapidly in Europe

324 (Stévant et al. 2017c) and biomass of this kelp species may be an alternative source of protein in food and feed

325 applications in the future. Not only the protein amount but also the protein quality is important when assessing

326 the nutritional value of a food product. The quality of a protein source is determined from its content in essential

327 amino acid (EAA, in mg amino acid g^{-1} protein) and compared to the EAA pattern of an ideal reference protein,

328 proposed by the WHO/FAO/UNU (2007). The tested protein is given a chemical score defined as the ratio between

329 each EAA of the protein source and the corresponding EAA level of the reference protein. Proteins from animal

330 sources generally have a chemical score of 100%, i.e. they contain all EAA in sufficient amount, while proteins

331 from vegetal sources (i.e. cereals, legumes, beans and nuts) have lower values due to at least one limiting EAA

332 (WHO/FAO/UNU, 2007). The levels of EAA in *S. latissima* samples in this study (tryptophan being excluded),

333 exceeded the minimum values required in human nutrition, resulting in chemical scores of 100%. A comparable

334 high chemical score (82%), with lysine as the first limiting EAA, was reported by Murata and Nakazoe (2001) for

335 the same species originating from Japan. However, the protein quality of cultivated *S. latissima* samples from

336 Denmark analyzed throughout a year was limited by low levels of histidine resulting in substantially lower

337 chemical scores (16.7% to 68.9%, Marinho et al. 2015). Despite remarkable amino acid profiles, the protein

338 digestibility of brown seaweeds is generally limited by the high content of dietary fibers and particularly the

339 alginate fraction in kelp species (Horie et al. 1995) as well as phenolic compounds (Wong and Cheung 2001b).

340 Lower levels of polyphenols were found in *Sargassum* spp. samples following air-drying at 60°C compared to

341 freeze-drying, which also resulted in significantly higher protein extractability and digestibility of the protein

342 concentrates in air-dried samples (Wong and Cheung 2001b). Although this aspect is not covered by the present

343 study, the levels of anti-nutritional factors generally limiting the digestibility of seaweed protein fractions (i.e.

344 alginate and polyphenols) were similar among treatment groups suggesting no effects of the tested drying

345 treatments on the digestibility of proteins from *S. latissima*.

346 It should be noted that the N-to-protein ratio of *S. latissima* measured in this study (3.98 ± 0.03 across sample

347 groups, n = 4) supports earlier results, reporting the inaccuracy of the commonly used conversion factor of N*6.25

348 to predict the protein content in brown macroalgae, due to the presence of non-protein N in the biomass (Angell

349 et al. 2016).

350

351 A biomaterial may undergo multiple chemical reactions upon drying e.g. browning reactions, lipid oxidation, and

352 protein denaturation, which can directly affect its quality. The present results did not reveal any major differences

353 among drying treatments with regard to the phytochemical content of *S. latissima* samples, in contrast with

354 previous studies (Chan et al. 1997; Wong and Cheung 2001a; Ling et al. 2015). However, losses of vitamins and

355 other bioactive secondary metabolites may occur during processing and storage of seaweed biomass (Lage-Yusty

356 et al. 2014), which were not estimated in the present study.

357 Mechanical alterations due to product shrinkage are also commonly observed from convective air-drying and

358 typically result in changes in the product shape and structure (Bonazzi and Dumoulin 2011). These alterations

359 may affect the extraction of phytochemical substances by influencing the factors governing solvent penetration in

360   the material e.g. capillarity, molecular diffusivity, which will ultimately affect their quantification. This should be

361   considered when studying the impact of a drying process on the chemical content of a biomaterial.

362

363   *Color and physico-chemical properties*

364   The hydration-related properties of plant materials such as WBC and SC, as well as OBC are related to the

365   chemical structure of their polysaccharides (Rupérez and Saura-Calixto 2001). Therefore, the alteration of these

366   parameters during the drying process can be the result of tissue damage. No significant differences in the WBC

367   of the samples were observed among treatments (table 4), which may also result from large variations among

368   sample replicates, particularly in air-dried samples at 25°C and 70°C. However, the WBC from air-dried samples

369   at 25°C were lower compared to the other treatment groups. The SC of the samples tended to decrease following

370   increasing drying temperatures and freeze-dried samples showed significantly higher SC compared to air-dried

371   samples. This can be explained by alterations of the textural properties of the biomaterial upon air-drying,

372   predominantly shrinkage, which have been reported in brown seaweeds (Cox et al. 2012; Sappati et al. 2017).

373   These mechanical alterations are resulting in changes in the microstructure of the product i.e. fewer pores and less

374   open structure, affecting the ability of the material to entrap water during rehydration. Product shrinkage is highly

375   dependent on the physical state (rubbery or glassy) of the material during the process. The effect of drying

376   temperature on the shrinkage of *S. latissima* was studied by Sappati et al. (2017) who measured greater rates of

377   shrinkage during air-drying at 70°C compared to 40°C. This was explained by a higher mobility of the solid matrix

378   during the process following higher temperature above the glass transition temperature (T_g). The drying

379   temperature during freeze-drying is typically below or close to T_g, maintaining the product in the glassy state

380   hence, minimizing the mobility of the matrix and subsequent shrinkage. The OBC measured in the *S. latissima*

381   samples was relatively high compared to the values reported by Rupérez and Saura-Calixto (2001) for other brown

382   seaweed species. Freeze-dried samples were also characterized by higher OBC values than air-dried samples. The

383   OBC of food products can be related to the levels of non-polar residues in the protein fractions (Chel-Guerrero et

384   al. 2002) and the nature of their polysaccharides (Fleury and Lahaye 1991) but also depend on other factors such

385   as the porosity of the material. In this study, no differences could be detected among samples neither regarding

386   their polysaccharides nor on their levels of non-polar amino acids (i.e. Gly, Ala, Val, Leu, Ile, Met, Phe and Pro,

387   Trp being excluded from the analysis). Hence, higher OBC of the freeze-dried samples is likely the result of a

388   more porous microstructure compared to air-dried samples.

389

390   The variations in the surface color among samples (defined by the coordinates $L*$, $a*$ and $b*$) were recorded using

391   computerized image analysis. The results, listed in table 4 were analyzed by PCA. The first two components of

392   the PCA biplot explained 97.6% of the variance (63.4% and 34.2% by PC-1 and -2 respectively, fig. 2) among

393   the data set. The variance in $b*$ (yellow/blue) explained by the first axis (PC-1), accounts for the largest part of

394   the total variance among samples, followed by $L*$ (lightness) and $a*$ (red/green). Graphically, sample groups can

395   be distinguished according to the $a*$ coordinate. Both samples air-dried at 25°C and freeze-dried exhibited a

396   predominant red hue ($a* > 0$) while green ($a* < 0$) was dominating in samples air-dried at 40 and 70°C (fig. 2 and

397   fig. S2 from online resource). Trends were also observed along $L*$ and $b*$, i.e. lighter freeze-dried samples and

398   increasing yellowness (increasing $b*$ values) with increasing drying temperatures, although the variability within

399   groups is high. Fucoxanthin (an orange pigment) is an important compound responsible for the coloration of

400   brown macroalgae but kelp species including *S. latissima* also contains other pigments such as violaxanthin and

401   β-carotene along with chlorophylls (Chl *a*, Chl *c*) (Haugan and Liaaen-Jensen 1994). Variations in color

402   characteristics among treatments may be the result of different reactions involving these pigments, leading to their

403   degradation or the formation of secondary colored substances. Similar results, i.e. decrease in $a*$ and increase in

404      $b*$ with increasing drying temperatures, were also reported by Moreira et al. (2016) in dried powder of *F.*

405   *vesiculosus*. These observations were explained by the authors by the leaching of the chlorophyll during the drying

406   process, resulting in increasing greenness, and its degradation, maximal within the range of 60 to 82°C, leading

407   to the yellowing of the material as carotenoids become more exposed.

408

409   ***Sensory properties and free amino acids***

410   The sensory characteristics including aroma, flavor, and texture qualities of *S. latissima* samples air-dried at 25˚C

411   and 70˚C were evaluated by eight trained panel members. No major differences were detected between the two

412   groups, based on the 13 selected sensory attributes listed in table 1 (fig. 3). The saltiness of the samples was

413   described as intense, which can be correlated with their particularly high ash levels. The samples were also

414   characterized by intense "fresh sea" aroma and flavor notes, while the umami flavor was only perceived as

415   moderate. The texture (i.e. mouthfeel) from both samples was neither perceived as cohesive nor crispy, neither

416   thin nor viscous and was rather tender. However, air-dried samples at 25°C dissolved more easily compared to

417   those dried at 70°C. This can be explained by the reduced porosity of the latter sample group, illustrated earlier

418   by lower SC due to product shrinkage. Although the intense perception of saltiness from the samples may have

419   affected the evaluation of flavor and texture, the results from this sensory evaluation are quite similar to those of

420 Chapman et al. (2015) who reported preliminary data from the sensory description of four edible seaweed species

421 including *S. latissima*.

422

423 Whereas there are numerous reports on the nutrient content of a wide range of seaweed species (see Holdt and

424 Kraan 2011, and references therein), few scientific studies have attempted to characterize the sensory profile of

425 relevant edible species. Some exceptions to this are studies describing the kelp *konbu* (*S. japonica*) as a rich source

426 of umami flavor, which is directly related to high levels of free glutamate, in its monovalent sodium-salt form

427 (monosodium glutamate, MSG) (Ikeda 2002; Ninomiya 2002). Generally, amino acids in their free form are

428 identified as major taste-active compounds in various foodstuffs. The free amino acid (FAA) composition of *S.*

429 *latissima* samples obtained from different drying treatments was analyzed and the results are summarized in table

430 5. The total amount of FAA represented approximately 8% of the total amino acids of the samples and did not

431 vary significantly among drying treatments. The samples contained high levels of alanine (perceived as sweet),

432 glutamate and aspartate in their free form (both eliciting umami sensation), relatively to other FAAs. The analysis

433 of the data by PCA only explained 77.6% (cumulated by PC-1 and -2) of the total variation in FAA and did not

434 provide an accurate comparison of the FAA profiles following different drying treatments. However, higher levels

435 of free glutamate were measured in freeze-dried and air-dried samples at 40°C (table 5). Although differences

436 were expected between samples air-dried at low (25°C) and high temperature (70°C) with regard to their

437 composition in aroma-active compounds, both groups displayed very similar FAA profiles correlating to their

438 similarity in flavor and aroma characteristics perceived during the sensory evaluation.

439

440 The sugar kelp *S. latissima* belongs to the same genus as Japanese *konbu*, however, the levels of glutamate

441 measured in this study, ranging from 1.03 to 1.52 mg g^{-1} DW, are far below the value reported by Ninomiya

442 (2002) for *konbu* (22.40 mg g^{-1} DW). Similarly, Mouritsen et al (2012) reported substantially lower amounts of

443 free glutamate, aspartate and alanine in broth extracted from *S. latissima* when compared to those from different

444 variants of Japanese *konbu*. After harvest, *konbu* is typically sun-dried and aged for several years in order to

445 develop characteristic flavors. The high content of free glutamate from aged *konbu* may result from the enzymatic

446 degradation of proteins during this maturation process. Sun-dried seaweeds typically contain higher moisture

447 contents compared to air- or freeze-dried material (Chan et al. 1997), which is an important factor governing

448 enzymatic activity. Endogenous enzymatic hydrolysis of proteins may occur during the storage of *konbu* leading

449 to high glutamate levels and characteristic umami flavor. Although Mouritsen et al. (2012) did not measure any

discernible effect of maturation (i.e. ageing of the dried product) in the glutamate content of *S. latissima* extracts, no mention is made of the drying technique used nor of the storage conditions (e.g. temperature, moisture content of the material) during the process. Optimizing storage conditions e.g. temperature and moisture, can be a key to develop preferable sensory profiles in edible seaweeds and future studies on this topic are envisaged. However, the sensory characteristics of seaweeds cannot be reduced to their FAA content since a wide range of molecules including peptides, minerals, low-molecular-weight carbohydrates and volatile compounds contribute to the sensory characteristics of foods (Lindsay 2008). The analysis of volatile oils from the steam distillation of several fresh edible kelp species from Japan identified a sesquiterpene alcohol, namely cubenol, as an important contributor to the kelp flavor (Kajiwara et al. 1988). López-Pérez et al (2017) identified 137 different volatile compounds in dried *S. latissima*, mainly consisting of (in decreasing order) carboxylic acids, hydrocarbons, alcohols, aldehydes, ketones and esters. In this comparative study on the aroma characteristics of 7 edible seaweed species in dehydrated form, a positive relationship could be established between the detected levels of volatile esters and hay aroma which was prominent in *S. latissima*. As reported by Michel et al (1997) high drying temperatures (i.e. 150°C), produce drastic changes in the composition of volatile compounds of dried *Ulva* sp. and *P. palmata* samples, as opposed to drying at lower temperatures (60°C), when compared to fresh samples.

Conclusion

Convective air-drying, especially at high temperatures affected the physico-chemical characteristics of *S. latissima*, compared to freeze-drying, used as a reference treatment in this study. Alterations were attributed to product shrinkage resulting in reduced porosity and rehydration capacity, potentially decreasing the quality and market value of the seaweed to be used as a functional ingredient by the food industry, or directly by the consumer in a rehydrated form. Aside from the iodine content which was significantly lower in freeze-dried samples, air-drying in the temperature range of 25 to 70°C resulted in equally nutritious products with similar flavor and aroma properties.

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483

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623 in vitro protein digestability and amino acid profile of protein concentrates. J Appl Phycol 13:51-58.

624 Yam KL, Papadakis SE (2004) A simple digital imaging method for measuring and analyzing color of food
625 surfaces. J Food Eng 61 (1):137-142.

626

627 **Table 1:** Sensory attributes, and their definitions, associated to the *S. latissima* samples.

Sensory attribute	Label	Scale anchors	Definition
Aroma			
Fresh sea	A – fresh sea	none ‖ much	Fresh sea odor
Fermented	A – fermented	none ‖ much	Fermented odor, pungent, marmite, matured cheese, cured
Hay	A – hay	none ‖ much	Dry hay, green tea
Flavor			
Salty	F – salty	none ‖ much	Salty taste
Fresh sea	F – fresh sea	none ‖ much	Sea flavor
Fermented	F – fermented	none ‖ much	Fermented flavor, matured cheese, marmite, cured
Hay	F – hay	none ‖ much	Fresh hay, green tea
Umami	F – umami	none ‖ much	Umami, meat stock, brown crab meat
Bitter	F – bitter	none ‖ much	Bitter taste
Texture			
Crispy	T – crispy	cohesive ‖ crispy	During first bites, how crispy is the sample
Chewy	T – chewy	tender ‖ chewy	While chewing, chewy: difficult to disintegrate
Viscous	T – viscous	thin ‖ viscous	Viscous, slimy, porridge like
Dissolves	T – dissolves	None ‖ much	Dissolves or melts easily in mouth while chewing

630 **Table 2:** Chemical composition of *S. latissima* samples air-dried at 25, 40, 70˚C and freeze-dried (FD). Values

631 are given as mean ± standard error (n = 3, unless stated otherwise). Different subscript letters in the same row

632 indicate significant differences (ANOVA, Tukey HSD, $p < 0.05$) among drying treatments.

	25˚C	40˚C	70˚C	FD
Residual moisture (%)	5.9 ± 0.2 [a]	4.4 ± 0.2 [ab]	4.0 ± 0.5 [b]	3.0 ± 0.4 [b]
Minerals				
Ash (% DW)	45.4 ± 1.7 [a]	45.2 ± 1.7 [a]	44.0 ± 2.2 [a]	43.8 ± 1.3 [a]
I (mg g^{-1} DW)	5.9 ± 0.3 [a]	5.7 ± 0.3 [a]	4.9 ± 0.2 [1 a]	3.0 ± 0.4 [b]
Carbohydrates (% Σ carb.)				
Alginate	54.6 ± 2.9	50.8 ± 2.3	53.6 ± 2.8 [1]	53.1 ± 1.4
Mannitol	25.5 ± 1.5	30.9 ± 2.0	28.0 ± 3.5 [1]	25.3 ± 3.5
Laminaran	7.1 ± 2.0	5.2 ± 1.4	4.9 ± 0.6 [1]	6.8 ± 2.5
Fucose (fucoidan)	4.7 ± 0.4	5.5 ± 0.5	5.4 ± 0.7 [1]	5.3 ± 0.2
Σ carbohydrates (% DW)	27.1 ± 0.5 [a]	27.8 ± 1.0 [a]	26.9 ± 0.2 [1 a]	23.9 ± 1.5 [a]
Polyphenols (% DW)	0.56 ± 0.03 [a]	0.67 ± 0.04 [a]	0.75 ± 0.02 [1 a]	0.63 ± 0.08 [a]
Fucoxanthin (mg kg^{-1} DW)	319 ± 49 [a]	na	737 ± 101 [1 a]	565 ± 115 [a]

633 [1] n = 2

634

635 **Table 3**: Amino acid composition (in mg amino acid g^{-1} protein), total amino acid (Σ AA, % DW), total essential

636 amino acid (Σ EAA, in mg amino acid g^{-1} protein), essential amino acid ratio (EAA/AA, dimensionless), and

637 chemical score (in %) of *S. latissima* samples air-dried at 25, 40, 70˚C and freeze-dried (FD). Values are given as

638 mean ± standard error (n = 3). Different subscript letters in the same row indicate significant differences (ANOVA,

639 Tukey HSD, $p < 0.05$) among drying treatments.

	25˚C	40˚C	70˚C	FD	Pattern [2]
Essential amino acids (EAA)					
Leu	89.3 ± 0.8	88.3 ± 0.5	87.4 ± 1.4	87.8 ± 1.8	63.0
Phe	63.7 ± 1.0	61.9 ± 0.8	59.9 ± 1.3	64.1 ± 1.5	46.0 [3]
Lys	59.2 ± 0.3	59.2 ± 1.1	59.2 ± 0.6	60.6 ± 0.5	52.0
Val	53.1 ± 0.4	53.0 ± 0.3	53.1 ± 0.5	52.6 ± 0.2	42.0
Ile	49.8 ± 0.5	49.6 ± 0.3	49.2 ± 0.5	48.9 ± 0.2	31.0
Thr	40.9 ± 1.5	39.9 ± 0.6	41.4 ± 1.7	40.7 ± 1.6	27.0
Met	24.1 ± 0.2	25.0 ± 0.3	24.7 ± 0.6	24.7 ± 0.4	26.0 [4]
His	21.6 ± 0.6	22.4 ± 0.3	20.8 ± 0.6	22.9 ± 0.5	18.0
Σ EAA	405.4 ± 1.0 [a]	403.0 ± 2.3 [a]	399.5 ± 6.9 [a]	405.3 ± 5.7 [a]	305.0
Chemical score (%)	100	100	100	100	

Non-essential amino acids
(NEAA)

Glu + Gln	120.0 ± 1.4	129.3 ± 2.8	125.6 ± 5.8	118.8 ± 2.6
Ala	117.9 ± 0.9	111.6 ± 4.6	120.5 ± 4.2	115.1 ± 3.0
Asp + Asn	90.0 ± 0.5	91.1 ± 4.1	87.4 ± 2.6	90.6 ± 1.9
Ser	58.9 ± 0.6	58.9 ± 3.2	53.9 ± 1.6	53.8 ± 2.6
Arg	51.8 ± 0.2	51.7 ± 0.5	50.9 ± 0.7	55.0 ± 0.4
Gly	50.8 ± 0.2	51.7 ± 0.4	50.5 ± 1.0	50.2 ± 0.3
Pro	46.8 ± 1.2	44.6 ± 1.3	49.2 ± 2.1	47.7 ± 1.4
Cys [1]	29.7 ± 0.4	27.7 ± 0.7	33.1 ± 2.2	31.3 ± 1.0
Tyr	27.2 ± 1.1	27.6 ± 0.9	26.8 ± 0.8	28.8 ± 0.6
Tau	1.5 ± 1.5	2.9 ± 1.5	2.7 ± 1.5	3.4 ± 3.4
Σ AA (% DW)	7.2 ± 0.3 [a]	7.3 ± 0.3 [a]	7.4 ± 0.3 [a]	7.2 ± 0.1 [a]
EAA/AA	0.4 ± 0.0 [a]	0.4 ± 0.0 [a]	0.4 ± 0.0 [a]	0.4 ± 0.0 [a]
Total N (% DW)	1.87 ± 0.10 [a]	1.85 ± 0.09 [a]	1.94 ± 0.05 [a]	1.82 ± 0.05 [a]
N-to-protein ratio	3.94 ± 0.07 [a]	4.03 ± 0.04 [a]	3.87 ± 0.03 [a]	4.03 ± 0.07 [a]

640   [1] quantified as cysteine, [2] EAA requirement pattern (WHO/FAO/UNU 2007), [3] Phe + Tyr, [4] Met + Cys.

641

642   **Table 4:** Physico-chemical properties and color parameters of *S. latissima* samples air-dried at 25, 40, 70˚C and

643   freeze-dried (FD). WBC and OBC are expressed in g water and g oil per g dried sample respectively, and SC is

644   expressed in ml per g dried sample. Values are given as mean ± standard error (n = 3). Different subscript letters

645   in the same row indicate significant differences (ANOVA, Tukey HSD, *p* < 0.05) among drying treatments.

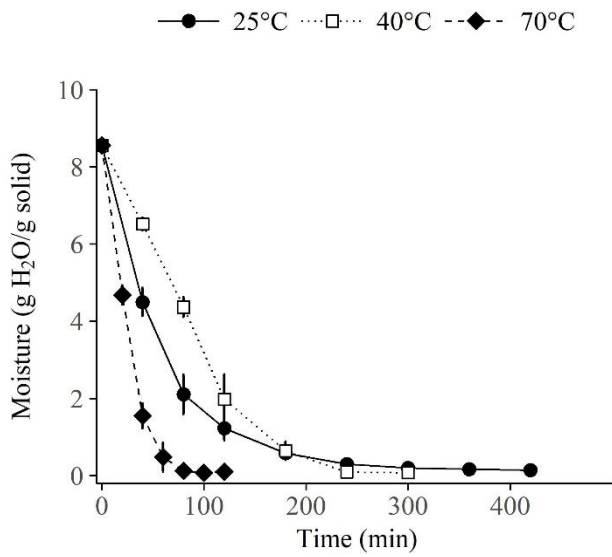|	25˚C	40˚C	70˚C	FD
Physico-chemical parameters				
WBC	6.7 ± 1.6 [a]	8.3 ± 0.6 [a]	7.3 ± 1.4 [a]	7.2 ± 0.2 [a]
OBC	4.1 ± 0.4 [a]	4.1 ± 0.1 [a]	4.4 ± 0.5 [a]	6.1 ± 0.1 [b]
SC	6.3 ± 0.5 [a]	5.0 ± 0.2 [a]	4.9 ± 0.1 [a]	10.2 ± 0.4 [b]
Color				
L*	46.0 ± 3.5	46.2 ± 3.9	52.3 ± 3.1	59.7 ± 2.5
a*	2.1 ± 0.5	-3.4 ± 0.9	-4.6 ± 1.8	2.1 ± 0.8
b*	34.0 ± 3.4	36.7 ± 4.8	44.3 ± 3.6	39.8 ± 3.5

646

647

648   **Table 5**: Total free amino acid content and free amino acid composition (in mg g^{-1} DW) of *S. latissima* samples

649   air-dried at 25, 40, 70˚C and freeze-dried (FD). Values are given as mean ± standard error (n = 3). Different

650   subscript letters in the same row (Σ FAA) indicate significant differences (ANOVA, Tukey HSD, *p* < 0.05) among

651   drying treatments.

	25˚C	40˚C	70˚C	FD

Free amino acids (FAA)

|				
Ala	2.07 ± 0.07	1.86 ± 0.13	2.07 ± 0.08	2.06 ± 0.15
Glu	1.03 ± 0.08	1.51 ± 0.03	1.22 ± 0.15	1.52 ± 0.06
Asp	1.02 ± 0.02	0.95 ± 0.16	1.04 ± 0.01	1.18 ± 0.08
Gln	0.32 ± 0.01	0.22 ± 0.02	0.33 ± 0.07	0.26 ± 0.02
Thr	0.32 ± 0.00	0.16 ± 0.02	0.20 ± 0.04	0.30 ± 0.04
Phe	0.17 ± 0.01	0.19 ± 0.01	0.24 ± 0.00	0.18 ± 0.01
Aba	0.12 ± 0.01	0.21 ± 0.01	0.23 ± 0.02	0.10 ± 0.02
Ser	0.13 ± 0.01	0.13 ± 0.01	0.15 ± 0.01	0.11 ± 0.00
Asn	0.11 ± 0.01	0.11 ± 0.01	0.12 ± 0.01	0.10 ± 0.01
Val	0.09 ± 0.01	0.10 ± 0.02	0.13 ± 0.03	0.08 ± 0.01
Lys	0.08 ± 0.01	0.08 ± 0.01	0.10 ± 0.01	0.07 ± 0.01
Tyr	0.08 ± 0.01	0.08 ± 0.01	0.08 ± 0.00	0.06 ± 0.01
Gly + Arg	0.07 ± 0.01	0.06 ± 0.01	0.08 ± 0.01	0.06 ± 0.01
Met	0.06 ± 0.00	0.06 ± 0.00	0.06 ± 0.00	0.07 ± 0.00
Leu	0.05 ± 0.01	0.06 ± 0.01	0.06 ± 0.01	0.04 ± 0.00
Ile	0.04 ± 0.01	0.04 ± 0.01	0.05 ± 0.01	0.03 ± 0.00
His	0.03 ± 0.00	0.03 ± 0.01	0.03 ± 0.00	0.02 ± 0.00
Σ FAA	5.8 ± 0.1 [a]	5.9 ± 0.1 [a]	6.2 ± 0.1 [a]	6.2 ± 0.2 [a]

652

653

Fig. 1: Experimental drying curves of *S. latissima* at 25, 40 and 70˚C. Values are given as mean ± standard error
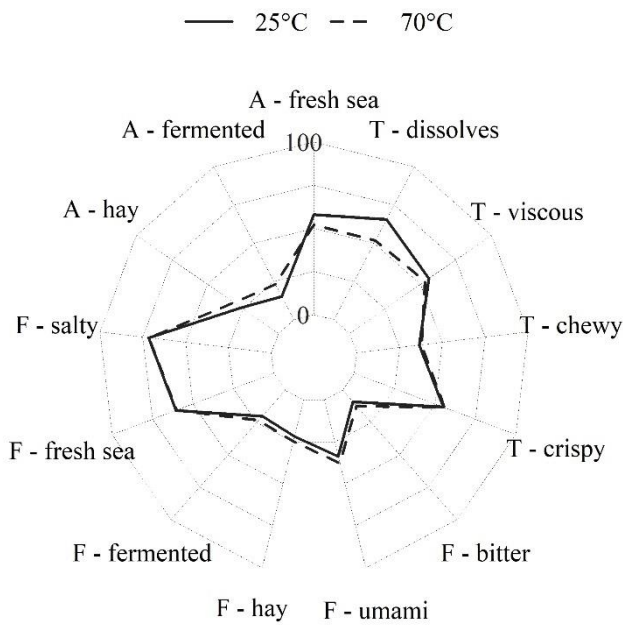
(n = 3).

654

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657
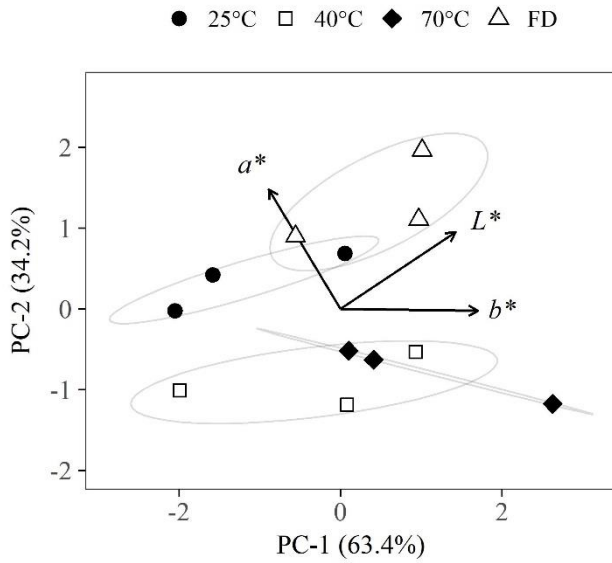
Fig. 2: PCA biplot (1st and 2nd principal component axis) obtained from the color analysis of *S. latissima* samples

659 air-dried at 25, 40, 70˚C and freeze-dried (FD). Vectors indicate loadings representing the variation in individual

660 color coordinates (*L**, *a** and *b**) among all samples.

661

662

Fig. 3: Mean values from descriptive sensory analysis of *S. latissima,* air-dried at 25 and 70˚C, including aroma

664 (A), flavor (F), and texture (T) characteristics.

Comments to reviewers and editor

Reviewer #1 & #2
Load density and sample mass in the dryer
Information on sample mass and density was added in lines 96-98.

Reviewer #2
Why using the monolayer configuration during drying?
By experience, stacking several seaweed fronds over each other may lead to uneven drying and case hardening. Although this configuration may not be appropriate in an industrial setting, it serves our purpose of studying the effects of drying temperature on the quality of the seaweed.

Reviewer #2
The indicated drying times (these were corrected in the new version of the manuscript) corresponded to the constant weight of the product. This detail was clarified in line 102 and drying times are mentioned in the first paragraph of the results and discussion (R&D) section.

Reviewer #2
Some additional data are interesting to add in order to improve the comprehension of the results.
Due to the amount of data from this experiment, the authors decided on splitting the results into two separate publications i.e. the present manuscript focusing on quality aspects of the material and a second on drying kinetics and technical aspects of the process. As the drying kinetics is an important factor influencing the quality of the final product, additional information was added to the manuscript in the first paragraph of the R&D section and in figure 1.

Reviewer #2
Relative humidity was constant, but what is the value?
This was corrected: RH was not constant upon drying and varied across temperatures. However, it was monitored during the process, added as supplementary online material to the manuscript and used in the discussion of the results (R&D section).

Reviewer #2
How long was the freeze-drying experiment?
line 239: "Freeze-drying of fresh *S. latissima* samples was achieved during a 20 h cycle."

Reviewer #2
How were the samples milled? Why 120 µm?
Biomass was milled using an electric grain miller. The 120µm powder grain size was chosen for the analysis to be comparable with that of Roleda et al. 2018 (see full reference in the manuscript's reference list)

Reviewer #2

"This result is expected due to lower drying capacities at lower temperatures". I consider that the result is more related to hygroscopic properties because at the same water activity the moisture content decreases increasing temperature.

An alternative explanation of this result based on the observed difference in RH between treatments was proposed in the first paragraph of the results and discussion section.

Reviewer #2

The comments about iodine content are interesting. Would be a vacuum drying an adequate technique to reduce iodine content for dried samples?

Although similar results were reported in the literature, other studies did not observe any effect of freeze-drying on the iodine content of seaweeds (see Nitschke & Stengel, 2016). As emphasized in this version of the manuscript these results should be interpreted with caution due to the small sample size and the reactive nature of iodide. Other thermal processes e.g. blanching will more effectively reduce the iodine content of kelps.

Reviewer #2

First paragraph of "color and physico-chemical properties": Perhaps a discussion based on processing time of seaweed elapsed at high temperature simultaneously at high moisture content would be interesting. Many reactions need simultaneously moisture content (plastifying effect) and temperature.

The possibility of longer drying times at 25°C resulting in increased pigment oxidation is already mentioned in the R&D section (line 295-304). Although the plastifying effect of water (i.e. even slight amount of water may considerably reduce T_g, increasing the rate of reactions in the matrix) is likely to affect the product quality upon drying, T_g was not measured in this study. An extensive discussion of the results from this perspective would be quite speculative.

Reviewer #2

First paragraph of "Sensory properties and free amino acids". Why were samples dried at 25 and 70ºC selected? Freeze dried samples would be useful as control…

The sensory evaluation of freeze-dried samples would definitely be useful and relevant. As mentioned in line 208 samples of *S. latissima* produced from different pre-treatments (4 in total) were evaluated in this sensory analysis and only the results from two of them (air-drying at 25 and 70°C) were discussed in this study. Since the evaluation of drying treatments on the organoleptic properties of *S. latissima* was not the main purpose of this session (the main purpose was the training of a sensory panel to the evaluation of seaweeds) the freeze-dried samples were left out of the analysis. Larger difference were expected between these two drying treatments but consistent results from the panel members demonstrated the opposite.

Reviewer #2

Second paragraph of this chapter: "The total amount of FAA represented… 8 and 9%...". Really? Are these values right? Please, verify, I have doubts analysing the results shown in Tables

This result was obtained from calculating:

Σ FAA / Σ AA * 100

Where Σ FAA is expressed in mg g^{-1} DW and Σ AA in % DW

From the analysis of the samples air-dried at 25ºC (values from tables 3 and 5), Σ FAA = 5.8 mg g^{-1} DW and Σ AA = 72 mg g^{-1} DW:

5.8/72*100 = 8.05%

Regarding the protein content (Σ AA) the results presented in this study are in the range of values reported in the literature (Schiener et al. 2016; Stévant et al. 2017b; see reference list in the manuscript). Although no reports of the FAA composition of *S. latissima* was found in the literature, the results from this study for Σ FAA are in the range of

those found earlier at the Norwegian University of Science and Technology (NTNU) for the same species (3 – 8.5 mg g^{-1} DW) and comparable to values found in other species i.e. *Alaria esculenta* (13 mg g^{-1} DW) and *Palmaria palmata* (20 mg g^{-1} DW) (unpublished results)

Reviewer #2

The conclusions section must be rewritten. Only conclusions based on the experiments must be written and no new ideas or discussions are necessary.
The conclusion was modified accordingly

Reviewer #2

Finally, the number of Figures and tables is too high (10). I suggest removing the first two Figures. The information given in the text is enough.
The PCA plots from AA composition and FAA composition were removed since they only partially explained the total variation among samples. The PCA plot from the carbohydrate composition was also removed as suggested.

Editor

Please reformat the references to the journal style (see attached style guidelines)
The manuscript (including the reference list) was adapted to the journal's guidelines. Supplementary online material was also added.

table 1

Table 1: Sensory attributes, and their definitions, associated to the *S. latissima* samples.

Sensory attribute	Label	Scale anchors	Definition
Aroma			
Fresh sea	A – fresh sea	none ‖ much	Fresh sea odor
Fermented	A – fermented	none ‖ much	Fermented odor, pungent, marmite, matured cheese, cured
Hay	A – hay	none ‖ much	Dry hay, green tea
Flavor			
Salty	F – salty	none ‖ much	Salty taste
Fresh sea	F – fresh sea	none ‖ much	Sea flavor
Fermented	F – fermented	none ‖ much	Fermented flavor, matured cheese, marmite, cured
Hay	F – hay	none ‖ much	Fresh hay, green tea
Umami	F – umami	none ‖ much	Umami, meat stock, brown crab meat
Bitter	F – bitter	none ‖ much	Bitter taste
Texture			
Crispy	T – crispy	cohesive ‖ crispy	During first bites, how crispy is the sample
Chewy	T – chewy	tender ‖ chewy	While chewing, chewy: difficult to disintegrate
Viscous	T – viscous	thin ‖ viscous	Viscous, slimy, porridge like
Dissolves	T – dissolves	None ‖ much	Dissolves or melts easily in mouth while chewing

table 2

Table 2: Chemical composition of *S. latissima* samples air-dried at 25, 40, 70˚C and freeze-dried (FD). Values are given as mean ± standard error (n = 3, unless stated otherwise). Different subscript letters in the same row indicate significant differences (ANOVA, Tukey HSD, $p < 0.05$) among drying treatments.

	25˚C	40˚C	70˚C	FD
Residual moisture (%)	5.9 ± 0.2 [a]	4.4 ± 0.2 [ab]	4.0 ± 0.5 [b]	3.0 ± 0.4 [b]
Minerals				
Ash (% DW)	45.4 ± 1.7 [a]	45.2 ± 1.7 [a]	44.0 ± 2.2 [a]	43.8 ± 1.3 [a]
I (mg g^{-1} DW)	5.9 ± 0.3 [a]	5.7 ± 0.3 [a]	4.9 ± 0.2 [1 a]	3.0 ± 0.4 [b]
Carbohydrates (% Σ carb.)				
Alginate	54.6 ± 2.9	50.8 ± 2.3	53.6 ± 2.8 [1]	53.1 ± 1.4
Mannitol	25.5 ± 1.5	30.9 ± 2.0	28.0 ± 3.5 [1]	25.3 ± 3.5
Laminaran	7.1 ± 2.0	5.2 ± 1.4	4.9 ± 0.6 [1]	6.8 ± 2.5
Fucose (fucoidan)	4.7 ± 0.4	5.5 ± 0.5	5.4 ± 0.7 [1]	5.3 ± 0.2
Σ carbohydrates (% DW)	27.1 ± 0.5 [a]	27.8 ± 1.0 [a]	26.9 ± 0.2 [1 a]	23.9 ± 1.5 [a]
Polyphenols (% DW)	0.56 ± 0.03 [a]	0.67 ± 0.04 [a]	0.75 ± 0.02 [1 a]	0.63 ± 0.08 [a]
Fucoxanthin (mg kg^{-1} DW)	319 ± 49 [a]	na	737 ± 101 [1 a]	565 ± 115 [a]

[1] n = 2

table 3 Click here to download table Table 3.docx ⬇

Table 3: Amino acid composition (in mg amino acid g^{-1} protein), total amino acid (Σ AA, % DW), total essential amino acid (Σ EAA, in mg amino acid g^{-1} protein), essential amino acid ratio (EAA/AA, dimensionless), and chemical score (in %) of *S. latissima* samples air-dried at 25, 40, 70˚C and freeze-dried (FD). Values are given as mean ± standard error (n = 3). Different subscript letters in the same row indicate significant differences (ANOVA, Tukey HSD, $p < 0.05$) among drying treatments.

	25˚C	40˚C	70˚C	FD	Pattern [2]
Essential amino acids (EAA)					
Leu	89.3 ± 0.8	88.3 ± 0.5	87.4 ± 1.4	87.8 ± 1.8	63.0
Phe	63.7 ± 1.0	61.9 ± 0.8	59.9 ± 1.3	64.1 ± 1.5	46.0 [3]
Lys	59.2 ± 0.3	59.2 ± 1.1	59.2 ± 0.6	60.6 ± 0.5	52.0
Val	53.1 ± 0.4	53.0 ± 0.3	53.1 ± 0.5	52.6 ± 0.2	42.0
Ile	49.8 ± 0.5	49.6 ± 0.3	49.2 ± 0.5	48.9 ± 0.2	31.0
Thr	40.9 ± 1.5	39.9 ± 0.6	41.4 ± 1.7	40.7 ± 1.6	27.0
Met	24.1 ± 0.2	25.0 ± 0.3	24.7 ± 0.6	24.7 ± 0.4	26.0 [4]
His	21.6 ± 0.6	22.4 ± 0.3	20.8 ± 0.6	22.9 ± 0.5	18.0
Σ EAA	405.4 ± 1.0 [a]	403.0 ± 2.3 [a]	399.5 ± 6.9 [a]	405.3 ± 5.7 [a]	305.0
Chemical score (%)	100	100	100	100	
Non-essential amino acids (NEAA)					
Glu + Gln	120.0 ± 1.4	129.3 ± 2.8	125.6 ± 5.8	118.8 ± 2.6	
Ala	117.9 ± 0.9	111.6 ± 4.6	120.5 ± 4.2	115.1 ± 3.0	
Asp + Asn	90.0 ± 0.5	91.1 ± 4.1	87.4 ± 2.6	90.6 ± 1.9	
Ser	58.9 ± 0.6	58.9 ± 3.2	53.9 ± 1.6	53.8 ± 2.6	
Arg	51.8 ± 0.2	51.7 ± 0.5	50.9 ± 0.7	55.0 ± 0.4	
Gly	50.8 ± 0.2	51.7 ± 0.4	50.5 ± 1.0	50.2 ± 0.3	
Pro	46.8 ± 1.2	44.6 ± 1.3	49.2 ± 2.1	47.7 ± 1.4	
Cys [1]	29.7 ± 0.4	27.7 ± 0.7	33.1 ± 2.2	31.3 ± 1.0	
Tyr	27.2 ± 1.1	27.6 ± 0.9	26.8 ± 0.8	28.8 ± 0.6	
Tau	1.5 ± 1.5	2.9 ± 1.5	2.7 ± 1.5	3.4 ± 3.4	
Σ AA (% DW)	7.2 ± 0.3 [a]	7.3 ± 0.3 [a]	7.4 ± 0.3 [a]	7.2 ± 0.1 [a]	
EAA/AA	0.4 ± 0.0 [a]	0.4 ± 0.0 [a]	0.4 ± 0.0 [a]	0.4 ± 0.0 [a]	
Total N (% DW)	1.87 ± 0.10 [a]	1.85 ± 0.09 [a]	1.94 ± 0.05 [a]	1.82 ± 0.05 [a]	
N-to-protein ratio	3.94 ± 0.07 [a]	4.03 ± 0.04 [a]	3.87 ± 0.03 [a]	4.03 ± 0.07 [a]	

[1] quantified as cysteine, [2] EAA requirement pattern (WHO/FAO/UNU 2007), [3] Phe + Tyr, [4] Met + Cys.

table 4

Click here to download table Table 4.docx ⬇

Table 4: Physico-chemical properties and color parameters of *S. latissima* samples air-dried at 25, 40, 70˚C and freeze-dried (FD). WBC and OBC are expressed in g water and g oil per g dried sample respectively, and SC is expressed in ml per g dried sample. Values are given as mean ± standard error (n = 3). Different subscript letters in the same row indicate significant differences (ANOVA, Tukey HSD, $p < 0.05$) among drying treatments.

	25˚C	**40˚C**	**70˚C**	**FD**
Physico-chemical parameters				
WBC	6.7 ± 1.6 [a]	8.3 ± 0.6 [a]	7.3 ± 1.4 [a]	7.2 ± 0.2 [a]
OBC	4.1 ± 0.4 [a]	4.1 ± 0.1 [a]	4.4 ± 0.5 [a]	6.1 ± 0.1 [b]
SC	6.3 ± 0.5 [a]	5.0 ± 0.2 [a]	4.9 ±0.1 [a]	10.2 ± 0.4 [b]
Color				
*L**	46.0 ± 3.5	46.2 ± 3.9	52.3 ± 3.1	59.7 ± 2.5
*a**	2.1 ± 0.5	-3.4 ± 0.9	-4.6 ± 1.8	2.1 ± 0.8
*b**	34.0 ± 3.4	36.7 ± 4.8	44.3 ± 3.6	39.8 ± 3.5

table 5

Table 5: Total free amino acid content and free amino acid composition (in mg g^{-1} DW) of *S. latissima* samples air-dried at 25, 40, 70˚C and freeze-dried (FD). Values are given as mean ± standard error (n = 3). Different subscript letters in the same row (Σ FAA) indicate significant differences (ANOVA, Tukey HSD, $p < 0.05$) among drying treatments.

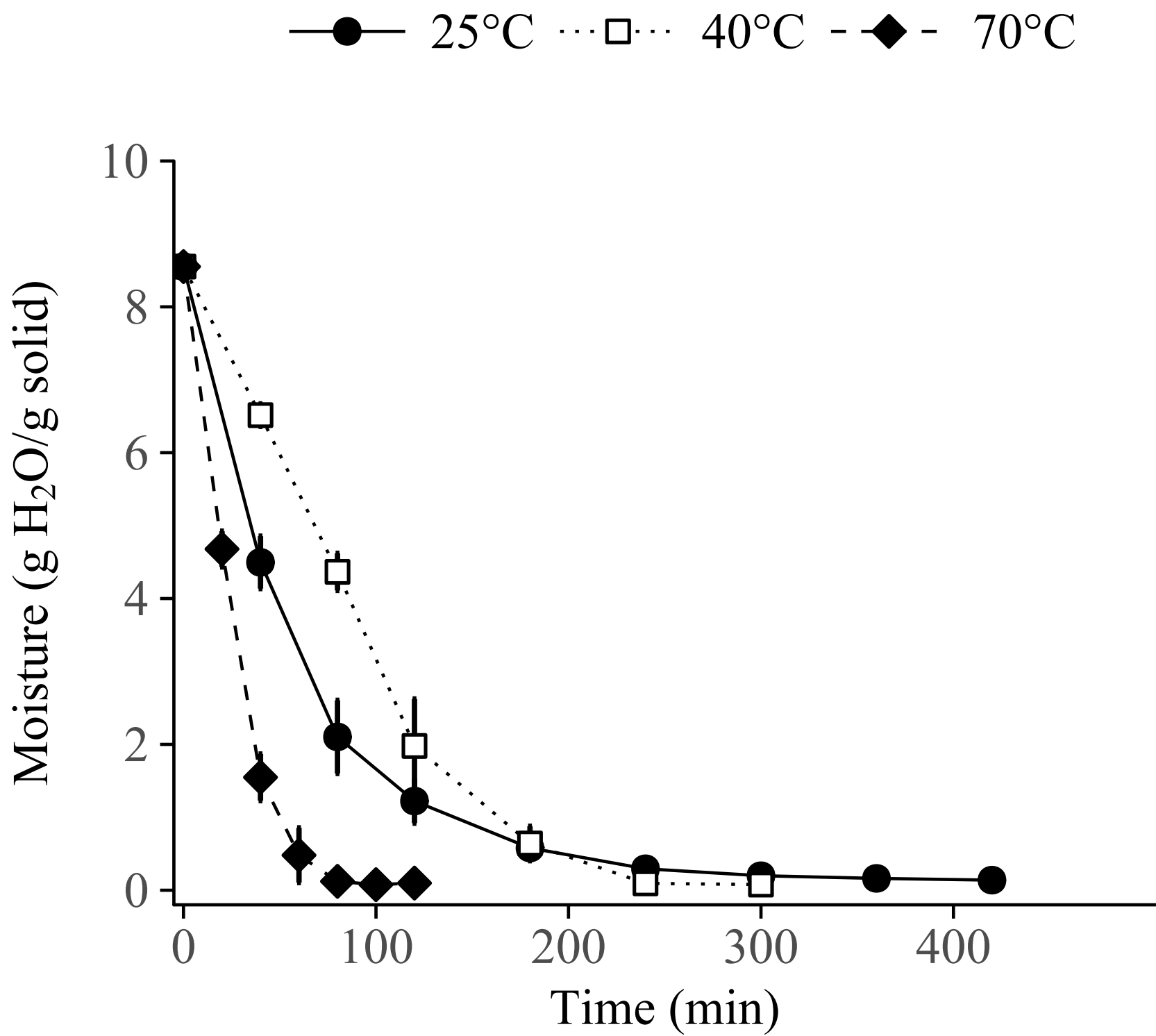|	**25˚C**	**40˚C**	**70˚C**	**FD**
Free amino acids (FAA)				
Ala	2.07 ± 0.07	1.86 ± 0.13	2.07 ± 0.08	2.06 ± 0.15
Glu	1.03 ± 0.08	1.51 ± 0.03	1.22 ± 0.15	1.52 ± 0.06
Asp	1.02 ± 0.02	0.95 ± 0.16	1.04 ± 0.01	1.18 ± 0.08
Gln	0.32 ± 0.01	0.22 ± 0.02	0.33 ± 0.07	0.26 ± 0.02
Thr	0.32 ± 0.00	0.16 ± 0.02	0.20 ± 0.04	0.30 ± 0.04
Phe	0.17 ± 0.01	0.19 ± 0.01	0.24 ± 0.00	0.18 ± 0.01
Aba	0.12 ± 0.01	0.21 ± 0.01	0.23 ± 0.02	0.10 ± 0.02
Ser	0.13 ± 0.01	0.13 ± 0.01	0.15 ± 0.01	0.11 ± 0.00
Asn	0.11 ± 0.01	0.11 ± 0.01	0.12 ± 0.01	0.10 ± 0.01
Val	0.09 ± 0.01	0.10 ± 0.02	0.13 ± 0.03	0.08 ± 0.01
Lys	0.08 ± 0.01	0.08 ± 0.01	0.10 ± 0.01	0.07 ± 0.01
Tyr	0.08 ± 0.01	0.08 ± 0.01	0.08 ± 0.00	0.06 ± 0.01
Gly + Arg	0.07 ± 0.01	0.06 ± 0.01	0.08 ± 0.01	0.06 ± 0.01
Met	0.06 ± 0.00	0.06 ± 0.00	0.06 ± 0.00	0.07 ± 0.00
Leu	0.05 ± 0.01	0.06 ± 0.01	0.06 ± 0.01	0.04 ± 0.00
Ile	0.04 ± 0.01	0.04 ± 0.01	0.05 ± 0.01	0.03 ± 0.00
His	0.03 ± 0.00	0.03 ± 0.01	0.03 ± 0.00	0.02 ± 0.00
Σ FAA	5.8 ± 0.1 [a]	5.9 ± 0.1 [a]	6.2 ± 0.1 [a]	6.2 ± 0.2 [a]

Figure 1

Figure 1

Figure 2

Figure 2

Figure 3

Figure 3
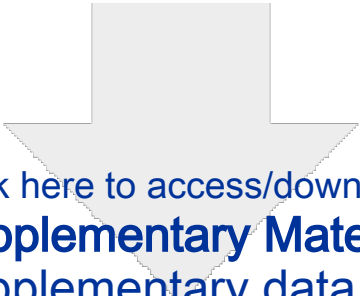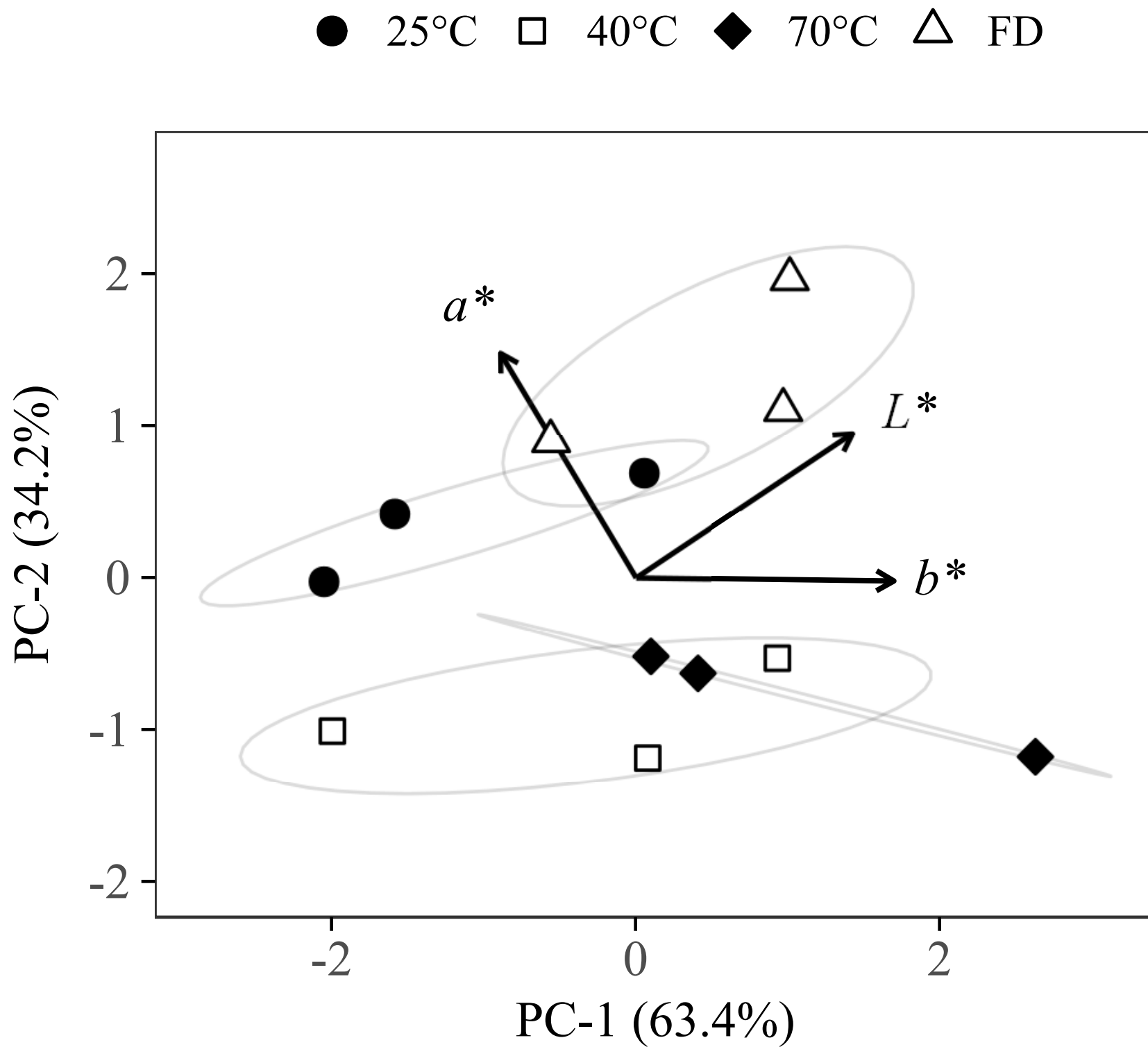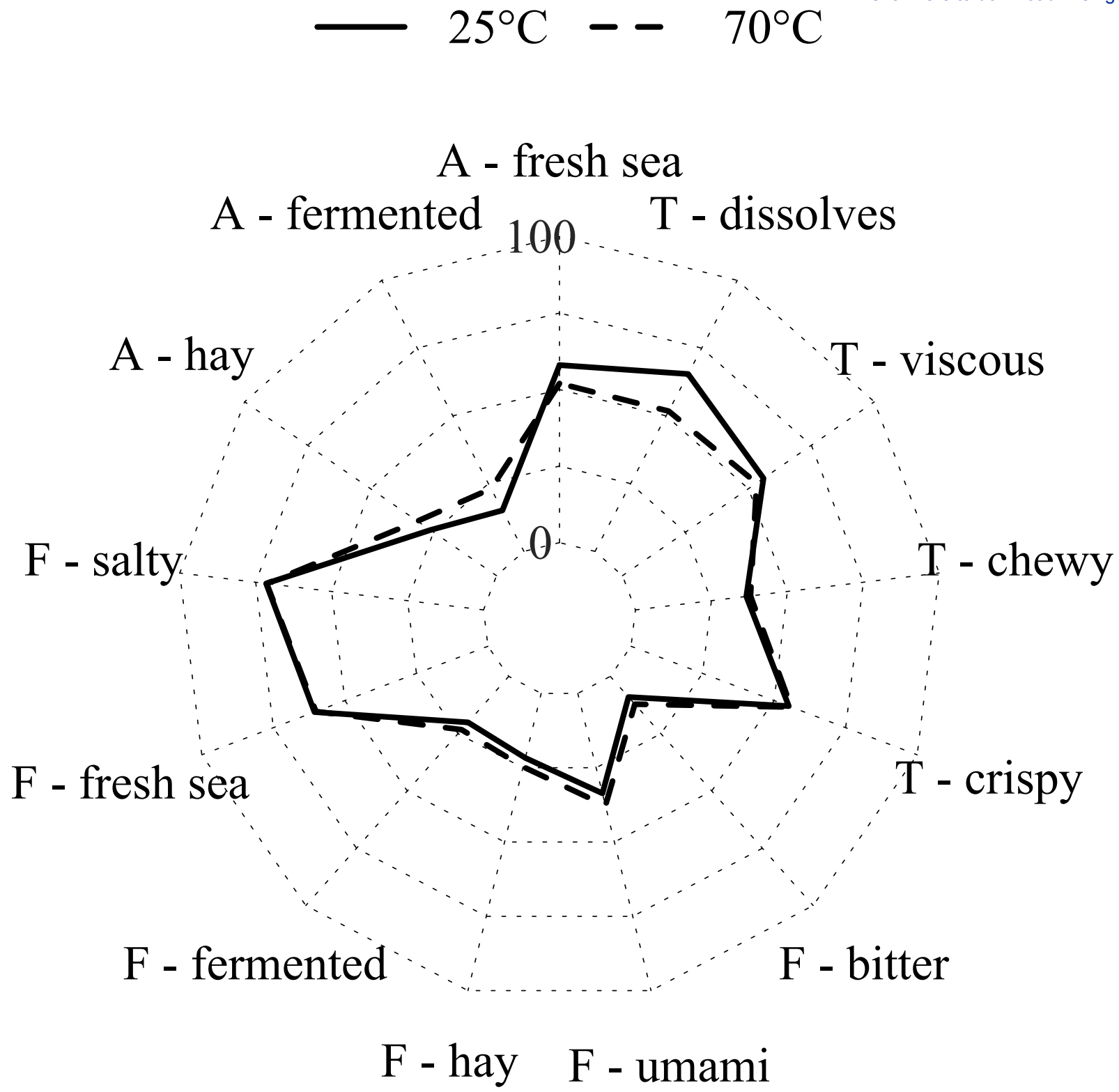
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Supplementary Material
Supplementary data.pdf