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PRELIMINARY STUDY ON PROCESSING SUGAR KELP (SACCHARINA LATISSIMA) USING SUPERHEATED STEAM DRYING AND STEAM PRE-TREATMENT

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SAMMENDRAG

Effektive preserveringsmetoder som minimerer tap av verdifulle komponenter og opprettholder kvaliteten er avgjørende for lønnsomheten og fremtiden for den voksende Norske næringen basert på tarebiomasse. Termiske prosesser som tørking kan effektivt stabilisere taren, men det er ofte store energikostnader og kapasitetsbegrensninger ved prosessering av store volumer. Makroterm prosjektet har undersøkt potensialet for å benytte overhetet damp- (OHD) teknologi til prosessering av sukkertarebiomasse (*Saccharina latissima*) som et alternativ til konvensjonell lufttørking. Tørkeforsøkene gjennomført i denne studien, samt data fra litteratur om OHD-tørking, antyder at denne teknologien kan gi redusert tørketid og energibehov samt bedre produktkvalitet (mindre produktkrymping, redusert jodinnhold) og dermed en mer bærekraftig prosess (mindre karbonutslipp) sammenlignet med lufttørking. Pilotutstyret som ble brukt under dette prosjektet hadde imidlertid begrensninger som resulterte i ujevn tørking og at fluidiseringsmaterialet festet seg på råstoffet, noe som fremhevet behovet for å tilpasse utstyret til tørking av tarebiomasse.

Under et eget forsøk ble jodinnholdet i *S. latissima* redusert ved eksponering mot damp. Resultatene fra denne testen antyder at dampbehandling som gir begrenset tap av oppløselige næringsstoffer, kan være et alternativ til blanchering.

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FORORD

Industriell utnyttelse av makroalger er i en rivende utvikling. Makroalger har mange bruksområder som mat og helseprodukter, fôr, gjødsel, biodrivstoff og kjemikalier. Norge har en lang kystlinje med en veletablert havbrukssektor som gir gode forutsetninger for utvikling av storskala dyrking av makroalger. En av hovedutfordringene for industrielle aktører er å ivareta kvaliteten på tarebiomassen etter høsting, ettersom store mengder biomasse høstes innenfor et begrenset tidsrom. Effektive preserveringsmetoder som minimerer tap av verdifulle komponenter og opprettholder kvaliteten på biomassen er derfor avgjørende for næringens lønnsomhet og fremtiden.

Makroterm prosjektets overordnede mål er å kartlegge om damp kan benyttes under prosessering av sukkertare til (i) tørking av biomasse med tørketeknologi basert på overhetet damp og (ii) damp som forbehandling for å redusere jodinnhold. Makroalger er et stort uutnyttet potensial for den norske havbruksnæringen. Nye effektive stabiliseringsmetoder vil sikre tilgang til tarebiomasse gjennom hele året, slik at industrien har bedre muligheter for å lykkes med kommersiell produksjon. Dette vil gi økt verdiskapning i kystnæringen og bidra til bærekraftig utvikling av norsk bioøkonomi basert på dyrking og prosessering av makroalger hvor region-Midt spiller en sentral rolle.

Prosjektet har vært nært koblet til det NFR-finansierte PROMAC-prosjektet, som blant annet fokuserer på energieffektiv prosessering av makroalger. Prosjektet ble ledet av Møreforsking Ålesund AS og gjennomført i samarbeid med SINTEF Ocean, Møreforsking Molde/NTNU, Seaweed Energy Solutions AS, Waister AS (tidligere MultiVector AS) og Tafjord Kraftvarme AS.

ABBREVIATIONS

AD	Air drying
ANOVA	Analysis of variance
DW	Dry weight
FD	Freeze-drying
GWP	Global warming potential
HPD	Heat pump drying
MC	Moisture content
NMKL	Nordic Committee on Food Analysis
RH	Relative humidity
RM ANOVA	Repeated measures analysis of variance
SS	Superheated steam
SSD	Superheated steam drying

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INTRODUCTION

The industrial utilization of macroalgae (also referred to as "seaweeds") is in rapid development in Europe, where the final products have several applications such as food and health, animal feed, manure, biofuels and chemicals. Seaweeds can be cultivated on a large-scale in coastal areas and may therefore offer superior alternatives to the production of terrestrial biomass with its related challenges, such as high demand on fresh-water resources. Norway has an extensive coastline and existing know-how on processing marine raw material and related infrastructure, thus, is a prime candidate for developing a bio-economy based on the cultivation and processing of seaweeds. Recent efforts from research, industry and public authorities have been committed to developing a seaweed industry in Norway and future perspectives for industrial development are positive (Stévant et al. 2017b). Currently, pilot-scale and pre-commercial seaweed farming projects largely focuses on kelp species, primarily *S. latissima* (fig. 1), due its phytochemical content and ability to achieve high biomass yields in short time (Broch et al. 2013; Handå et al. 2013; Wang et al. 2013).

One of the main challenges for industrial players is to preserve the biomass since seaweeds are characterized by a high moisture content and a rapid microbial decomposition once harvested. Encrusting fouling by bryozoans leads to extensive losses of biomass and considerable quality deterioration, forcing producers to harvest in May-June, before the onset of fouling (Forbord et al. 2012; Førde et al. 2016). Therefore, substantial volumes are typically harvested within a limited time frame in kelp cultivation (approximately 4-6 weeks), setting standards for efficient processing strategies (Stévant et al. 2017b). Preservation methods that (i) minimize losses of valuable compounds, (ii) ensure product safety and (iii) limit the energy use and associated costs, are a key to increase profitability of the industry.

Drying is the most common method for the stabilization of wet biomass including macroalgae. Drying is a mass transfer process that removes moisture from the product and reduces the water activity, thus preserving the product by avoiding microbial growth and limiting the rate of chemical reactions. In addition, the weight and volume of the material are substantially reduced, minimizing the packaging, storage and transportation costs. Primary processes of harvested seaweeds using convective air-drying can effectively stabilize the biomass but require technology and may be difficult to implement close to harvesting sites. Moreover, drying large biomass volumes is extremely energy demanding, lowering the environmental and economical sustainability of the process chain (van Oirschot et al. 2017) and may affect the nutrient content (Chan et al. 1997; Tello-Ireland et al. 2011; Moreira et al. 2016; Chenlo et al. 2017) and the physicochemical properties of the material (Stévant et al. 2018).

Superheated steam drying (SSD) is gaining priority in the food processing industry as a potential replacement technology to increase the energy efficiency of drying processes. This technology involves the use of recirculated superheated steam (SS) instead of hot air, as drying medium to supply heat to the product and remove the evaporated water. The higher capacity and thermal

conductivity of SS leads to higher drying rates (Mujumdar 2014). The exhaust superheated steam can be used as a heat source to improve the energy efficiency of the system. In addition, nutrient losses due to oxidation are avoided and mechanical stress resulting in e.g. product shrinkage is limited. This technology applied to drying kelps was tested by Jia et al. (2018), who reported a reduction of the energy input by nearly 50 % from a SSD system bearing exhaust heat recovery unit, compared to a convection air-dryer. The present work reports on the energy requirements associated to the process and its environmental impact. In Europe, large amounts of surplus heat are available from various industrial processes and integrated models using this secondary energy source are suggested for seaweed processing (Philis et al. 2018).

Although seaweeds are known to be a rich source of nutritious and bioactive substances, they may also accumulate toxic elements with potentially negative effects on human health. In the case of *S. latissima*, iodine present in excessive amounts may be a problem in the context of using this species in human nutrition (Lüning and Mortensen 2015; Desideri et al. 2016; Stévant et al. 2017a). Heat treatments using fresh water (blanching) reduces considerably the iodine content of *S. latissima* (Lüning and Mortensen 2015) but also results in losses of water-soluble nutritional compounds, such as minerals and carbohydrates. This detrimental effect may be avoided by using steam instead of hot fresh water to reduce the iodine content. Hence, the effect of steam exposure on the iodine content of *S. latissima* was investigated in this study.



Fig. 1: Sugar kelp (Saccharina latissima)

MATERIAL AND METHODS

Seaweed raw material

Biomass of *S. latissima* was harvested at Seaweed Energy Solutions (SES) at Frøya, Norway, in May 2018, then transported within a few hours to the processing plant (HitraMat), where the seaweeds were stored in large tanks (2 m³) provided with seawater circulation (9 °C). This biomass entered two separate experiments studying the effects of i) SSD on the quality of *S. latissima* as food and ii) a steam pre-treatment on the iodine content of the samples.

Superheated steam drying

SSD of *S. latissima* was performed using a batch system in an automated fluidized-bed test dryer (BioWaste 20, Waister AS, Norway) using superheated steam (SS) as drying medium. In this system, the steam is circulated and superheated in a semi-closed loop system. While a part of the SS leaving the drying chamber is superheated and reintroduced into the drying chamber as drying medium, the remainder passes through a condenser for the removal of evaporated water out of the system (fig. 2). The pressure inside the drying chamber was close to atmospheric pressure. Wet raw material was fed into the dryer by a screw conveyer with a cutting mechanism. However, to ensure appropriate and uniform particle size *S. latissima* was manually pre-cut.

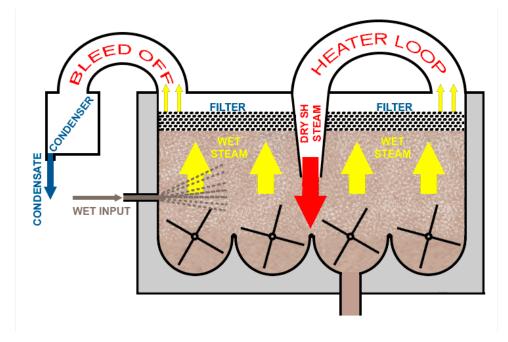


Fig. 2: Schematic illustration of the BioWaste 20 SSD system

Wood chips (1.0 to 2.0 kg) was first fed to the system as mechanical fluidizing medium to prevent the adhesion of the wet biomass to the walls, paddles and shafts inside the drying chamber. Subsequently, 10 kg of fresh *S. latissima* was fed to the system. The relative humidity (RH), SS temperature to the drying chamber and the outlet temperature were constantly monitored. The product temperatures and moisture levels were monitored at regular intervals. The power consumption from heater, mixers and fan, displayed on the dryer, was recorded during each test.

During the first phase of the process, SS at a maximum temperature of 120 °C was sent to the drying chamber until the RH in the chamber reached 70 %. To avoid a drastic increase in product temperature, the maximum SS temperature to the chamber was set to 85 °C during the final phase of the process. Drying was stopped when the RH in the chamber reached 55 %, as preliminary tests under these conditions showed this level to be indicative of a dried seaweed product. The moisture content of the seaweed product during drying was measured with an infrared moisture analyzer (Mettler Toledo MJ33). The dried product consisting of seaweeds and wood chips was then discharged and vacuum-packed for further manual separation. Simultaneously, samples were vacuum-packed and frozen for subsequent freeze-drying (FD). Both SSD and FD samples were analyzed for their chemical content.

Three batch tests were performed, where the ratio of seaweed/wood chips was adjusted. Additionally, the SSD system was tested as a single replicate, in a semi-continuous mode under the same conditions as described previously, where 42 kg of fresh seaweeds were added to the system and fed to the drying chamber at regular time intervals.

Steam blanching pre-treatment

Blades of *S. latissima* were placed in a compartment of a benchtop food steamer (Phillips HD 9140). Samples were taken prior to and after 1, 3 and, 10 min treatments, vacuum-packed and frozen until FD, for further analysis of the iodine content. The treatment was performed in 3 replicates.

Chemical analyses

Moisture – The final moisture content (MC) in the dried samples was determined gravimetrically by drying at 105 °C until constant weight of the samples was achieved (typically 24 h). The subsequent results from chemical analyses were expressed as part of the dry weight (DW) of the samples.

Ash content – The ash content was determined after combustion of the dried samples at 590 °C for 12 h in a laboratory muffle furnace. The ashes were quantified gravimetrically as the residue from combustion and expressed as % DW.

Protein content – The organic nitrogen content was quantified by the Kjeldahl method (NMKL 6 2003) and an estimate of the total protein content was calculated by multiplying the nitrogen content by a factor of 4 as previously reported suitable to predict the protein content of *S. latissima* harvested in the same area in May (Stévant et al. 2018).

Carbohydrate content – The total carbohydrate content of the samples was estimated as follows:

% Carbohydrate = 100 - % Ash - % Proteins - % Lipids

Lipid content – Pulverized dried samples were hydrolyzed with hydrochloric acid (HCl) to release bound lipids. The lipids were then extracted after addition of alcohol, dimethyl-ether and petroleum-ether and determined gravimetrically after complete evaporation of solvent.

Iodine content – The determination of iodine in the samples was based on the colorimetric Sandell-Kolthoff-reaction depending on the reduction of cerium (IV) sulfate by arsenite in the presence of iodide (Yaping et al. 1996). Dried seaweed samples were burned at 1000 °C, to convert all inorganic and organic iodine species to iodide (I⁻). The residues were solubilized in deionized water and sodium arsenite (NaAsO₂) was added to the solutions in microplates. After the addition of cerium (IV) sulfate Ce(SO₄)₂ and shaking, the microplates were allowed to stand for 20 min away from ambient light. The absorbance of the remaining Ce (IV), representing the amount of iodine in the samples, was measured in a spectrophotometer at 436 nm and compared to a standard curve.

Statistical analyses

All statistical analyses were performed on R software (R Development Core Team 2018) including functions from the nlme package (Pinheiro et al. 2017). Raw data were pre-processed for descriptive statistics and the results expressed as mean \pm standard error (n = 3, unless stated otherwise). An analysis of variance (ANOVA, R function aov) was used to detect differences in individual quality parameters among SSD- and FD-samples. A repeated measures ANOVA (RM ANOVA, R function lme) was used to detect differences in the iodine content of steamed samples over time.

Environmental impact from drying seaweeds

The environmental impacts in this project are calculated using the GaBi LCA Software, in combination with the accompanying Professional Database 2018. The model used to calculate the impacts, was built to represent the system studied, including direct energy consumption and infrastructure. The input data for the model is a combination of upscaled energy measurements and relevant generic data from databases. The main assumptions in terms of data used, is the process of upscaling. A combination of literature data and experience with upscaling is used here.

RESULTS AND DISCUSSION

Experimental superheated steam drying of S. latissima

The process of drying *S. latissima*, harvested from a commercial cultivation site in Norway and initially containing $92.1 \pm 0.0 \%$ (n = 16) water, using SSD was investigated. This technique consists in using steam at a temperature higher than its boiling point, which then becomes superheated, as a medium to carry moisture out of the product. SS is in many ways a better drying medium than air due to its physical properties, heat and mass transfer, as well as more efficient penetrability (Mujumdar 2014). The heat transfer coefficient of steam is twice that of air. The viscosity (penetrability) of steam is at the same time almost half of the viscosity of air. Therefore, SSD has the potential to shorten drying time and reduce the energy demand considerably compared to air drying.

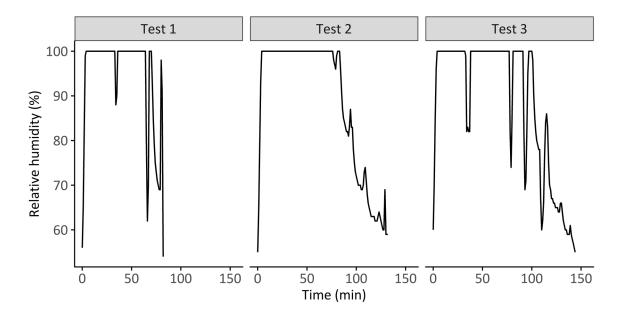


Fig. 3: Experimental drying of *S. latissima* using SSD. The product was considered dry when the RH in the chamber reached 55 %. The ratio seaweed/wood chip (w/w) was 10/2 in test 1and 10/1.5 in tests 2 and 3.

The drying time, from the three tests performed was between 80 and 150 minutes (fig. 3). A direct comparison with another drying technology was not conducted during this preliminary study. However, Sehrawat et al. (2016) reports higher drying rates using SSD technology compared to conventional convection air-drying systems at temperatures above 100 °C (at atmospheric pressure). This suggests that the rate of SSD at the temperatures used in this study (SS

temperature in the range 80 to 110°C) would be slightly higher or similar than for air-drying at the same temperatures. Relatively low temperatures (below 80 °C) are usually employed to dry seaweeds to limit the destruction of heat-sensitive substances and maintain product quality. The product temperature measured during the process did not exceed 54 °C. An increase in the temperature of the product and negative effects on quality may occur during the last phase of the drying where equilibrium moisture of the product is achieved. At this stage, the energy from the medium is no longer transferred to the moisture for evaporation leading to the elevation of the product temperature.

Heterogeneity was observed in the MC of the product within each test i.e. some fragments of kelps were dried while other contained higher amounts of water. This can be due to the seaweed material attaching to the elements of the drying chamber, and/or to self-adhesion and formation of clusters limiting further drying of the product. However, the MC of representative dry samples was measured at the end of each test and was 6.2, 3.1 and 3.2 % in test 1, 2 and 3 respectively. The seaweeds attached to the wood chips, used as fluidization material, and could not be easily separated after the drying process. Complete optimization of the system including feeding of the wet biomass, design of the drying chamber and fluidization medium is necessary to avoid these problems and provide a homogeneous dry product.

Energy requirements of drying S. latissima using SSD technology

The energy use was measured during the experiments using the internal sensors in the SS dryer. Data was collected before and after each test. The energy consumption measured was 14 kWh in test 1, 19 kWh in test 2 and 21 kWh in test 3. The differences among each test are explained by differences in drying times.

Using the result from test 1, in which the seaweed biomass was dried to 6.2 % MC, the total energy consumption given above corresponds to 1.5 kWh kg⁻¹ evaporated water, or 12.7 kWh kg⁻¹ dried product (containing 6.2 % moisture). This energy consumption per kg dried product is relatively high and can be attributed to the pilot scale dryer in this experiment, which is not specifically designed for drying seaweed biomass. For an industrial steam drier that dry to 10 % water content it should be possible to have an energy use of 4-5 kWh/per kilo dried algae. **TABLE 1** shows the advantage of SSD compared to other drying methods in terms of energy requirements.

Drying method	Energy consumption	
Air-drying 40 °C ¹	7.27	
Air-drying 70 °C ¹	7.20	
Superheated steam drying (SSD)	5	
Vacuum freeze drying (FD) 1	17	

Table 1: Energy consumption (in kWh kg⁻¹ dried product) associated with different industrialdrying methods

¹ Data from Nordvedt et al. (unpublished results from the PROMAC project)

Effects of SSD on the quality of S. latissima

The chemical composition of *S. latissima* dried using SSD technology was analyzed and compared to the quality of FD samples. Generally, due to the absence of liquid water and the low temperatures during the process of freeze-drying (FD) biomaterials, the rate of most reactions responsible for product deterioration are very low, resulting in high quality food products (Bonazzi and Dumoulin 2011). However, because of the high equipment and operating costs of FD, air drying is often preferred and regarded as viable method to process significant amounts in a shorter time. The MC of SSD samples indicated in table 2 is higher than the levels measured immediately after drying, mentioned above, because of the necessary separation of the seaweed fragments and fluidization material (wood chips) and the resulting water reabsorption during the operation.

SSD samples were characterized by lower ash and iodine contents compared to FD samples. Reducing the iodine content of kelps is considered a positive effect since this essential element in excessive amounts is identified as a potential risk for human health in the context of using seaweeds, particularly kelp species, in food applications (Stévant et al. 2017a). However the value measured in SSD samples still exceeded the limit value of 2000 mg kg⁻¹ DW established by the French food safety authority for seaweed food products (CEVA 2014). Iodine from seaweeds can be reduced in contact with hot water (Lüning and Mortensen 2015) even at moderate temperature levels (Stévant et al. 2017a). The reduction of water-soluble compounds such as iodine and ashes (reflecting the mineral content) can be due to the condensation of vapor over the surface of the feed material at the initial stage of SSD, reported as a common problem in SSD operations (Sehrawat et al. 2016). However, further investigations using an adapted dryer must be conducted to properly assess the effects of SSD on water-soluble compounds of kelps.

	SSD ^a	FD	ANOVA (p-value)
MC ^b	10.7 ± 0.4	7.5 ± 0.8	0.051
Ash ^b	38.7 ± 2.7	48.9 ± 1.8	0.047
Carbohydrates ^b	47.3 ± 2.8	39.1 ± 1.7	0.071
Lipids ^b	4.1 ± 0.3	2.5 ± 0.2	0.014
Proteins (N * 4) ^b	9.9 ± 0.3	9.5 ± 0.3	0.55
lodine ^c	3581 ± 544	5690 ± 173	0.020

Table 2: Chemical composition of superheated steam dried (SSD)- and freeze dried (FD)-samples of *S. latissima*. Significant ANOVA results are indicated in bold.

 $a_n = 2$

^b in g (100 g)⁻¹ DW

^c in mg kg⁻¹ DW

The protein content remains constant, likely due to their association with cell-wall polysaccharides, as previously observed during soaking treatments of *S. latissima* in fresh water (Stévant et al. 2017a). A lower lipid content in FD samples can be explained by a long-term frozen storage of the samples before FD (4 months), and possible enzymatic lipid oxidation during this period.

In comparison, a recent study comparing the effects of air-drying at temperatures ranging from 25 to 70 °C to FD on the quality of *S. latissima* did not measure significant differences in the chemical composition of the samples among treatment groups (Stévant et al. 2018).

Due to the adhesion of wood chips to the seaweeds, the analysis of the physico-chemical properties of the samples, i.e. water- and oil-binding capacity (WBC and OBC) and swelling capacity (SC), could not be conducted. As reported by Stévant et al. (2018) air-drying, especially at high temperatures (70 °C) leads to the alteration of these properties, due to product shrinkage and reduced porosity during the drying process. However, the inspection of the *S. latissima* samples following SSD, a high porosity of the dried products was observed suggesting that limited mechanical stress is applied to the product upon SSD resulting in low rates of shrinkage and high hydration related properties of the product. These observations support those from previous studies showing SDD to be an effective drying technology to maintain the quality (texture, microstructure, retention of bioactive compounds) of food products (Sehrawat et al. 2016).

The fucoxanthin content of the samples was also analyzed but was below the detection limit (0.1 mg kg⁻¹) in all samples most likely due to the extended storage period prior to analysis due to the

necessary separation of the seaweeds from the wood chips. Evidences from an earlier study clearly show the sensitivity of fucoxanthin during storage (Indrawati et al. 2015).

Environmental impacts drying S. latissima using SSD technology

When looking at the environmental impacts from drying seaweed in a life cycle perspective, there are in general two main elements to consider: direct energy consumption, and the infrastructure needed for the drying. The functional unit used to assess the drying, is one kg of dried seaweed containing 10 % moisture. The results in global warming potential (GWP) are illustrated in fig. 4. The direct energy consumption has a significantly bigger impact than the infrastructure. The impact associated to the energy input of SSD technology is 32 % less than that of conventional convective air-drying methods and 71 % less than the energy requirements of FD. In this case, the direct energy consumption comes from the Norwegian electricity grid mix, consisting mainly of electricity from hydro power (96.2 %) as well as natural gas (1.83 %) and wind power (1.56 %). The remaining energy sources (0.41 %) comes from of hard coal, coal gases, heavy fuel oil, biomass, biogas and waste incineration.

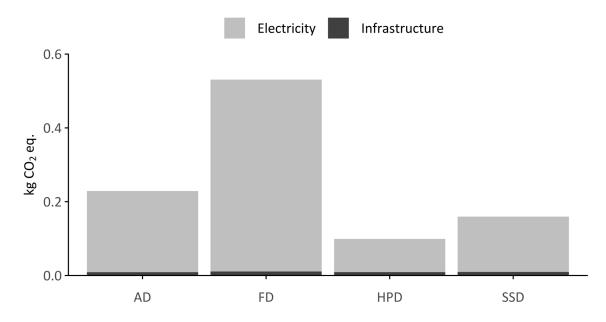


Fig. 4: GWP per kg dried seaweed following different drying technologies i.e. air drying (AD), freeze drying (FD), heat pump drying (HPD) and superheated steam drying (SSD).

When looking at the energy consumption associated to a process, the environmental impact in terms of GWP from different energy sources can be compared. The fig. 5 shows the impacts of SSD of seaweeds, using energy from the Norwegian electricity grid mix, hydro- and wind

electricity. We see that shifting the energy source to more renewable energy sources will have significant impact on the GWP per kg seaweed dried.

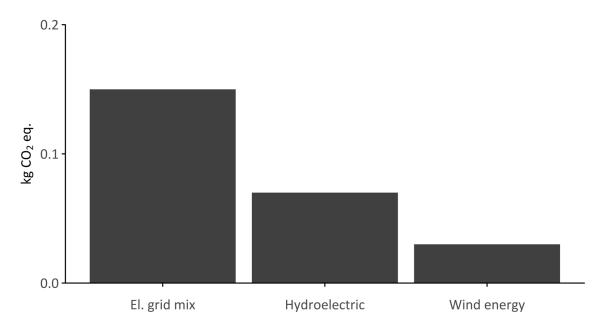


Fig. 5: GWP per kg seaweed from superheated steam drying (SSD) technology using different energy sources.

Steam blanching pre-treatment of S. latissima

The use of steam as an initial pre-treatment of *S. latissima* was investigated in this study with the aim of reducing the iodine content of the biomass and improve its quality as a food ingredient and limiting losses of water-soluble compounds.

The temperature measured in the compartment of the benchtop steam cooker used in this study was 95 °C. A significant reduction of the iodine content of the samples was measured during the treatment (fig. 6, RM ANOVA, p = 0.005). However, the iodine level was not reduced to below the threshold value of 2000 mg kg⁻¹ DW after 10 min. Although the chemical analysis of water-soluble compounds was not conducted in this preliminary study, the DW of the fresh steamed and control samples at t₀ was in the range between 8.1 and 9.1 % (wet weight basis) and did not vary significantly during treatment (RM ANOVA, p = 0.314) suggesting that the nutrient composition of the samples remained relatively stable.

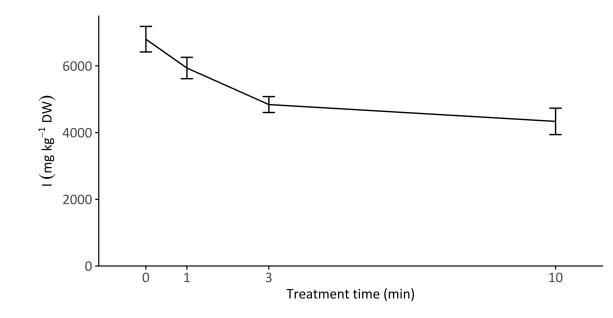


Fig. 6: Iodine content of *S. latissima* during steam blanching treatments.

CONCLUSIONS

The potential of SSD technology for the large-scale processing of *S. latissima* was investigated in this preliminary study. The experimental drying conducted in this study as well as general data on SSD of biomaterial reported in the literature, suggests the competitive advantage of this technology in terms of processing time, energy requirements and carbon emissions. However, the limitations of the pilot equipment used during this project, resulting in uneven drying and attachment of the fluidization material to the seaweeds, highlighted the need to optimize the dryer.

Analysis of the SSD samples suggests that this technology produces high-quality seaweed products suited for multiple industrial application and characterized by lower rates of shrinkage during the process (high porosity of the product) compared to conventional air-drying methods and the retention of bioactive substances. Future work will focus on optimizing this technology and investigate further the advantages of using these products in food applications. Increasing the processing efficiency of seaweed raw material will benefit a growing industry producing and using this sustainable resource in Norway and increase sustainability of the value-chain.

Lower iodine levels were measured in SSD samples compared to FD samples which is positive in the context of using this kelp species in food applications. In a separate experiment, steam treatments significantly reduced the iodine of *S. latissima* and may be an alternative to blanching limiting losses of soluble nutrients.

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