LOW TEMPERATURE DRYING AND THERMO-PHYSICAL PROPERTIES OF BRONW SEAWEEDS (SACCHARINA LATISSIMA)

I. Tolstorebrov[1], T.M. Eikevik[1], M. Bantle[2]

[1]*Norwegian University of Science and Technology*
Department of Energy and Process Engineering
Kolbjorn Hejes vei 1B, NO-7491 Trondheim, Norway
Email: ignat.tolstorebrov@ntnu.no
[2]*SINTEF Energy Research, Department of Thermal Energy*
Kolbjorn Hejes vei 1B, NO-7491 Trondheim, Norway

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ABSTRACT

The drying kinetics of brown seaweed *Sacharina Latissima* with and without blanching were investigated in temperature range between 10.0 and 38.0 °C. The drying process is more rapid for raw seaweeds. The effective moisture diffusivity coefficient (with respect to shrinkage) was found in the range between 0.5 and 5.0 10^{-10} m^{-2}/s. Drying temperature influence on lightness and hue color parameter of the seaweeds. The salt content in raw seaweeds influence on the shape of the sorption isotherm at high relative humidifies. The differential scanning calorimetry of raw seaweed samples revealed glass transition at ultra-low temperature range, while it was not found for blanched seaweeds. State diagram for drying and freezing of raw seaweeds was made.

INTRODUCTION

The concept of food from seaweed becomes an important issue today. It fits perfectly well in a frame of sustainable management of marine resources. The global market for seaweed is approximately 30 million tonnes with a value of 6-7 billion EUR (95 % of the production is from cultivation in Asia), Kappahycus alvarezi, Japanese kelp (Laminaria japonica), Gracilaria spp. and Wakame (Undaria pinnatifida) being the major seaweed species (FAO, 2014).
(FAO, 2016). The 36.0 % of seaweed was used as a direct food source, and the rest as a food additive and chemical products (hydrocolloids, feed, fertilizers and high value ingredients) (FAO, 2014). Laminaria japonica (Kombu) and Saccharina latissima (Royal Kombu) was the most popular seaweed as a food (Chopin, 2012).
The key advantage (and the main proposal) of seaweed industry in Europe is its independence from three main resources, that limit conventional agriculture: land water and fertilizers (Radulovich, 2011). The long coastline of Europe and different climate zone makes the seaweed cultivation diverse and sustainable.

Drying is one of the common ways of doing it. Most of the seaweeds are sold in a dried form (Forster & Radulovich, 2015). Drying is used for stabilization of seaweed as a final product or as a material for further processing (extraction of bioactive substances: proteins and lipids, purification etc.).
The different methods of solar and natural drying are utilized today. This is the cheapest method, however, the dried product has a high amount of water content at the end of the process (up to50.0 % dry basis) (Phang, Chu, Kumaresan, Rahman, & Yasir, 2015). This is explained by high water absorption properties of the seaweeds and high relative humidity of the ambient air. The natural drying process usually takes 5 or 7 days (Fudholi et al., 2011). Such traditional way of seaweed drying at open air refers to the times when seaweed was used as a raw material for hydrocolloid industry, feed additives and fertilizers. It is further not feasible and safe to apply the same method for food graded seaweeds in the climate conditions of the most productive cultivation sites in Northern Europe.
The problems of traceability and safety appear, when the drying process is used for food production needs. The drying of seaweeds for long time and/or high temperature influences negatively on their carbohydrates, amino acid composition and vitamin content (Chan, Cheung, & Ang, 1997). The main requirements are:

- Stable and gentle temperature and humidity regime. The increasing of drying temperature will reduce the drying time, but will significantly reduce the polysaccharide content, some components can also decompose;
- Short time of dehydration is also essential due to enzymatic activity(Gupta, Cox, & Abu-Ghannam, 2011);
- Low temperature drying or freeze drying for seaweeds, which will be used as a material for extraction(Hahn, Lang, Ulber, & Muffler, 2012; Pangestuti & Kim, 2015).

The task of this study is determination of the main dependencies for the low temperature drying of seaweeds with the aim to implement these knowledge for design of conveyor drier.

MATERIALS AND METHODS

Characterization of raw material

The raw material a brown algae (commonly referred as a kelp) specie *Saccharina latissima* (Sugar kelp or kombu). The seaweeds are the one-year old aquaculture product, which is produced in Norway, Sør-Trondelag. The brief characteristic of the aquaculture seaweeds is given in the Table 1.

Table 1. Chemical composition of *Saccharina latissima* at harvesting season *(source SES internal database)*

Parameter	*Saccharina latissima*
Protein, g/100g d.w.	13.2
Fat, g/100g d.w.	2.9
Carbohydrates, g/100g d.w.	55.3
Dietary fiber, g/100g d.w.	46.6
Ash, g/100g d.w.	37.9
Water, %	89.7
Energy, kcal/100g d.w.	301.0

The kelp came to NTNU in a frozen state (November 2016-February 2017). The seaweed was frozen and packed in 2.0 kg bags with a high barrier properties. The moisture content was measured at 89.8 (0.3) %, when the seaweeds were dried at 105.0 °C without any processing. However, squeezing the water helped to increase the solid content to 14.0-15.0 % w.b.

Description of drying experiments

The seaweeds were divided into two groups one was raw seaweeds (without any processing) another was blanched seaweeds. The blanching took place in a boiling water (100.0 ºC) for 1.0 min. The following parameters were varied during drying experiments:

- Air temperature: 10; 25 and 38 ºC;
- Type of drying: on shelves and hanged;
- Thickness of seaweed layer: 1 layer, 2 and 3. The seaweeds were placed in a drying chamber on shelves parallel to the air flow.
- Air relative humidity for all cases was in the range between 12 and 20 %, air velocity 1- 2 m s^{-1}.

DSC analysis

The DSC analysis was done with a DSC Q 2000 (TA instruments, USA) equipped with a Liquid Nitrogen Cooling System. Samples were cooled and equilibrated for 5 minutes at –150.0 °C; the cooling rate was 10.0 °C min^{-1}. Then the samples were heated up to 150.0 °C (depending on moisture content) with the heating rate of 10.0 °C min^{-1}.

The glass transition, freezing and melting temperatures and unfreezable water determination

The incipient melting point (“end of freezing”), T'_m, was detected by analyzing the DSC heating curve (I. Tolstorebrov, T. Eikevik, & M. Bantle, 2014). The freezing point was estimated as the minimum value of the ice melting endothermic peak on the DSC heat flow curve.
The glass transition was determined with TA Universal Analysis 2000 version 4.5A software (TA instruments, USA). The glass transition is characterized with the following parameters: the onset (T_{go}), end (T_{ge}) and inflection (T_{gi}) points. It should be noted, that the glass transition in seaweeds is relatively weak. Thus, the inflection point was determined as a negative peak of the derived heat flow curve. The amount of unfreezable water was detected by the DSC melting curve analysis, (I. Tolstorebrov, T. M. Eikevik, & M. Bantle, 2014)

Statistical analysis

The analysis of variance (ANOVA: single test and two-factor test with replication) was applied to analyze the obtained data. The difference was considered significant at $p<0.05$.
A regression analysis was done with a software DataFit 8.1 program (Oakdale Engineering). The quality of the regression was evaluated with the following parameters: F-Ratio, Prob(F) and R^2. F-Ratio is the ratio of the° mean regression sum of the squares divided by the mean error sum of the squares. Prob(F) is the probability that the null hypothesis is true. R^2 is the coefficient of multiple determinations. The standard deviation is introduced in the brackets after the values given in the text.

RESULTS AND DISCUSSION

Influence of pretreatment and drying modes on color of seaweeds

The blanching process alternated the lightness (L*), chrominance (C), and hue (h) of the seaweeds ($p<0.05$). The typical brown color of the seaweeds visually changed to green, this was reflected by increasing of hue by 12° alteration of chrominance by 5 points, Table 2. The green color appeared by washing out or by decomposition of fucoxanthin due to the bleaching process. The other pigment chlorophyll is more stable to such treatment.
The dried seaweeds showed other dependence. The lightness of raw samples were significantly higher when compared with blanched ($p<0.05$). This happened due to deposits of salts of the surface of the dried blades of Saccharina Latissima. Chrominance of raw seaweeds was also higher ($p<0.05$). Some interesting observations were found for influence of temperature on the hue of the

dried seaweeds. The increasing of temperature to 38.0 °C resulted in slight decreasing of hue, so the color became more “yellow” (in terms of L*C*h* color space), while the seaweeds dried at 10.0 °C showed more “green” colour. This was valid both for raw and blanched seaweeds.

Table 2. Color parameters of raw and blanched seaweeds before and after drying

Type of seaweeds	**Before drying**								
	Color parameters								
	L*, (-)		**C, (-)**		**H,º**		**a*, (-)**		**b*, (-)**
Raw seaweeds	9,78 (0,96)		8,08 (1,97)		80,23 (1,45)		1,39 (0,43)		7,96 (1,94)
Blanched seaweeds	11,54 (1,29)		13,84 (1,63)		92,00 (4,04)		-0,13 (1,25)		14,74 (1,39)
After drying									
	L*, (-)			**C, (-)**			**H,º**		
	38.0ºC	**25.0ºC**	**10.0ºC**	**38.0ºC**	**25.0ºC**	**10.0ºC**	**38.0ºC**	**25.0ºC**	**10.0ºC**
Blanched seaweeds	23,1^{a}	23,7^{a}	23,83^{a}	6,4^{c}	5,76^{c}	6,59^{c}	81,39^{e}	82,85^{e}	86,86^{f}
	(1,54)	(2,19)	(1,23)	(2,84)	(1,89)	(1,32)	(5,66)	(4,57)	(3,3)
Raw seaweeds	25,86^{b}	27,25^{b}	27,91	8,71^{d}	8,39^{d}	8,42^{d}	83,39^{e}	86.00^{f}	86,73^{f}
	(1,12)	(2,78)	(2,46)	(1,33)	(1,02)	(1,09)	(3,07)	(2,53)	(3,03)

Sorption isotherms of seaweeds

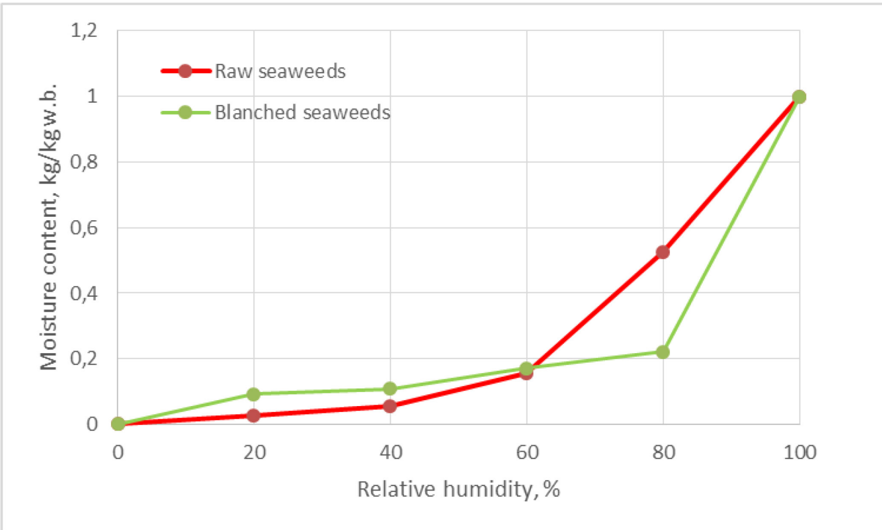

Figure 1. Sorption isotherms for raw and blanched seaweeds at 25.0 ºC.

The experiments to determine the influence of relative humidity of the drying air of the drying kinetics were done, Figure 1. Blanched seaweeds showed higher accumulation of moisture at low relative humidity of the drying air (up to 60.0 % RH), when compared with raw seaweeds.

The increasing of the relative humidity of the drying air resulted in a sharp increasing of moisture content in raw seaweeds, while blanched seaweeds showed linear trend of moisture increasing in the range between 20.0 and 60.0 % RH. These dependencies and trends were observed for all the investigated temperatures of drying air.

The sharp increasing of the equilibrium moisture content at 80.0 % and relatively low water absorbance at RH below 60.0 % can be explained by hygroscopic point of *NaCl*, which is appeared at 75.5 % RH at 25.0 C.

Drying kinetics of seaweeds

The drying kinetics was investigated for raw and blanched seaweeds. These raw seaweeds showed initial moisture content at 900.0 % d.b., which is relatively high when compared with other types of products suspected to convection drying. The high moisture content can be explained by the age of the seaweeds: they were harvested at the first year of growth on aquaculture site. Such type of

seaweeds is aimed for direct consumption in salads, snacks, soups etc. In addition, the blades of luminaria had small thickness and absence of any parasites or sedimentations.
The bleaching process resulted in a significant decreasing of dry matter. Thus, the moisture content of the blanched seaweeds was found at 2079.0 % d.b.

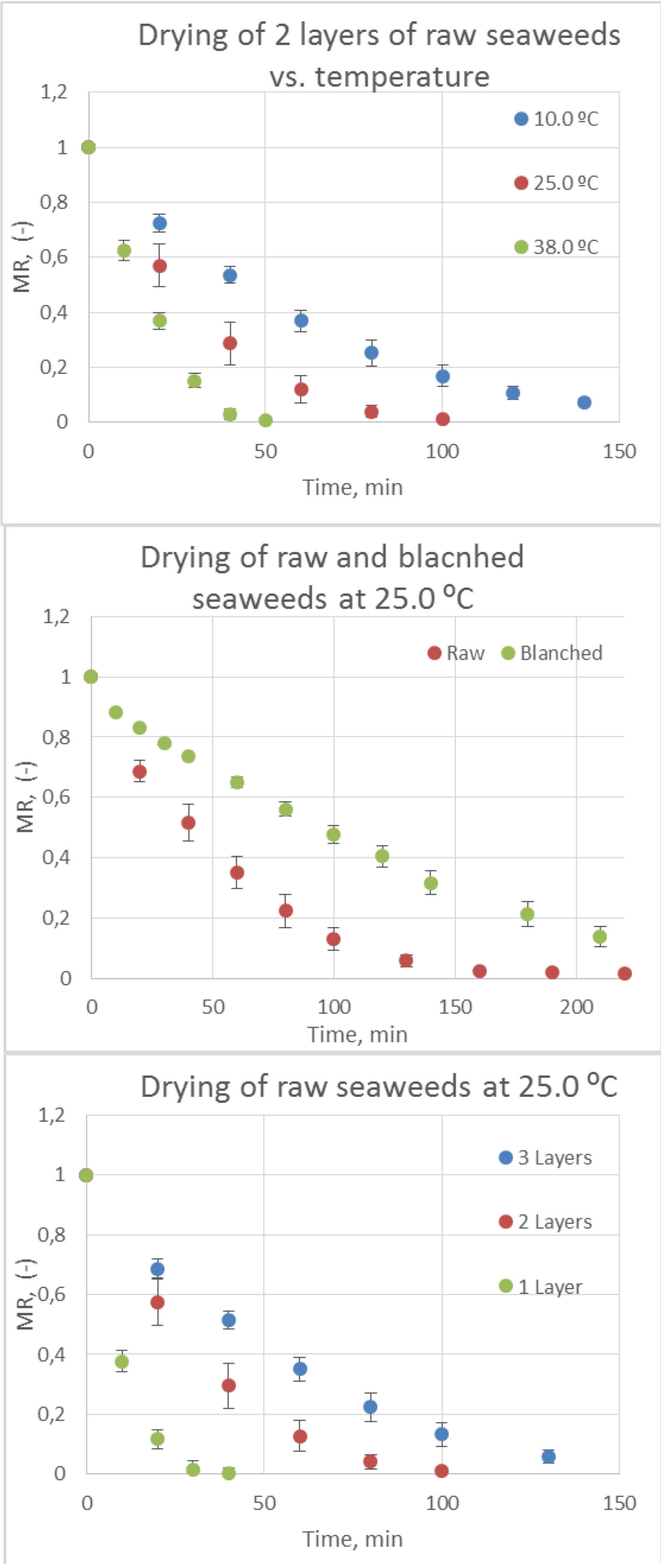

Figure 2 Example of drying kinetics for seaweeds

Preliminary drying experiments revealed that the freeze drying of seaweeds takes long time without any positive influence on structure and color of the dried product, when compared with drying at low temperatures in the range between 10 and 38.0 °C. The examples of drying kinetics (with standard deviations) in the temperature range between 10.0 and 38.0 °C are shown on Figure 2.
Dewatering of raw seaweeds was much faster, when compared with blanched. This was explained by the higher moisture content of blanched seaweeds and changes of their structure after belching. The increasing of temperature accelerated the dewatering of the seaweeds and the highest dewatering rate was observed for 38.0 °C.
The seaweeds were organized into flat layers on the shelves to understand the influence of layer thickness on drying kinetics. It was remarkable, that the difference in drying rate for 1 and 2 layers were very small in some experiments, especially for blanched seaweeds. This happened due to the specific structure of the seaweed's blades, when the thickness varied from the edges to the middle part of the seaweeds in the range between 0.08 to 0.25 mm. However, the increasing of number of the layers slow down the drying process significantly. The drying of 5 layers of the seaweeds took inappropriate long time for the conveyor type of drying at a given temperature range.
The drying behaviors were modelled using Newton model, as soon as the thickness of raw seaweed was in the range between 0.1 and 0.2 mm, equation:

$$MR = \exp(-k\tau) \qquad (1)$$

where k - drying constant , min^{-1}.
The regression equations were obtained for all the investigated temperatures

drying regimes. All the models showed high quality (R^2>0.98; Prob(F)<0.000014; F(Ratio) >700). The empirical drying coefficient k was found in the range between 0.013 and 0.0077 due to influence of temperature and thickness of the sample. At the same time, assuming that the drying temperature influences the same way on the drying kinetics, these coefficient *k* were normalized with respect to thickness on the sample. Thus, the Equation 2 can be introduced in a such way:

$$MR = \exp\left(-\frac{k_{norm}}{n}\tau\right) \quad (2)$$

where n is dimensionless empirical coefficient which is related to the amount of layers of seaweeds (or thickness of the layer).

Table 3 introduces the empirical drying coefficients for Equation 2. The coefficient n equal 4 for 2 layers of raw seaweeds, which is equivalent to square of thickness. At the same time, n was found to be 6 for 3 layers of seaweeds, this can be explained by relatively high deviation of the seaweed blade thickness, shrinkage and development of moisture gradient inside the 3 layers. The drying coefficient for blanched seaweeds was determines at 1, 2 and 4 for one, two and three layers respectively. This shows that even short-term treatment changes the nature of the material and its composition. As it can be seen from Table 3 that the drying constant is increasing with the increasing of the temperature and its value is 2-3 times higher for raw seaweeds, when compared with blanched.

Table 3. Coefficients for Newton equation, seaweed drying

Type of seaweeds	Temperature, °C	k, min^{-1}	n, (-)		
			1 layer	2 layers	3 layers
Raw seaweeds	10.0	0.07 (0.003)	1	4	6
	25.0	0.13 (0.05)			
	38.0	0.22 (0.05)			
Blanched	10.0	0.035 (0.04)	1	2	4
	25.0	0.051 (0.04)			
	38.0	0.060 (0.05)			

The more precise way to characterize the drying behaviors is determination of the effective moisture diffusivity. This material property can be derived from the drying kinetics via analytical solution of the Fick's second law of diffusion (infinite slab), equation:

$$MR = \frac{8}{\pi}\sum_{n=0}^{\infty}\frac{1}{(2n+1)^2}\exp\left(\frac{-(2n+1)^2\pi^2 D_{eff}\tau}{4(h)^2}\right) \quad (3)$$

where D_{eff} – effective moisture diffusivity , $m^2\ s^{-1}$; τ – drying time, sec; h – half-thickness of seaweed layer, m; n – number of terms (as positive integer, 35 for this study).

The model includes thickness of the product, which is changing within drying. The shrinkage should be considered in the case of seaweeds due to their high moisture content, especially blanched seaweeds. The decreasing of the thickness can be expressed by simple regression equation:

$$h = a * x_{d.b.}^{n} \quad (4)$$

where a and n are empirical parameters a=0.0678 and 0,0687 and n=0.3582 and 0.3614 for raw and blanched seaweeds respectively. The thickness of *Laminaria Saccharina* blades varied from 0.25 mm (0.05) to 0.08 (0.03), which creates some difficulties for mathematical description of the drying process. The thicker part situated in the middle of a blade (leaf) and the thinner areas are on the edges. However, the average trend of thickness decreasing was used in this study. Knowing the change of thickness with respect to moisture content, the Equation 4 can be used to calculate effective moisture diffusivity with respect to shrinkage, Equation 5 (Arévalo-Pinedo & Murr, 2006):

$$\frac{Y_\tau - Y_e}{Y_0 - Y_e} = \frac{8}{\pi} \sum_{n=0}^{\infty} \frac{1}{(2n+1)^2} \exp\left(\frac{-(2n+1)^2 \pi^2 D_{eff.shrk} \tau}{4(h)^2}\right) \tag{5}$$

where $D_{eff.shrk}$ – effective moisture diffusivity with shrinkage, $m^2\ s^{-1}$; Y is a ratio of moisture content, X kg kg^{-1} d.b., to volume of the product; the shrinkage of the surface of the seaweeds during drying was considered to be negligible, thus, the thickness of the samples (Equation 4) were used.
Table 4 introduces the determined effective moisture diffusivity for different temperatures. This property of material was determined separately for 3 different thicknesses of the product with respect to shrinkage.

Table 4. Effective moisture diffusivity (D $m^2\ s^{-1}$ $*10^{10}$) of seaweed with respect to shrinkage, temperature and type of pre-treatment

Type of pre-treatment	Nr. of layers	Temperature of the drying air, °C		
		10	25	38
Blanched seaweeds	1	1.00	1.80	1.50
	2	1.00	2.60	4.00
	3	1.50	2.60	5.00
	Average	**1.17 (0.28)^{a}**	**2.33 (0.46)^{b}**	**3.5 (1.8)^{b,c}**
Raw seaweeds	1	0.50	1.10	1.50
	2	0.90	1.50	3.60
	3	0.90	1.10	2.60
	Average	**0.77 (0.23)^{a}**	**1.2 (0.23)**	**2.60 (1.1)^{c}**

The average values of effective moisture diffusivity increased with increasing of the drying temperature and reached the highest value of $3.5*10^{-10}$ m/s^{-2} at 38.0 °C. The effective moisture diffusivity of blanched seaweeds were relatively lower, when compared with the raw. At the same time there were statistical difference between D for raw and blanched seaweeds at 10.0 and 38.0 °C, due to high standard deviations of results. Usually, salted product show lower moisture diffusivity coefficient, when compared with unsalted. We suppose, that in our case osmotic dehydration influenced the process, due to low thickness of the product. Previous study of drying of Irish brown seaweeds revealed the effective moisture diffusivity of $12.2*10^{-07}$ m^2/s (Gupta et al., 2011), which much higher normal range of moisture diffusivity between 10^{-9} and 10^{-12} m^2/s (Onwude, Hashim, Janius, Nawi, & Abdan, 2016).

Glass transition and freezing points of seaweeds with different moisture contents

Knowledge of phase transition in a product at different temperatures is very important for design drying equipment, choosing packaging material and storage conditions. The freezing point and ice content can show amount of solid fractions during freeze-drying process. Also, it can give information about stickiness of the granules and other structural changes during drying.
The glass transition was found for all the samples of raw seaweeds, Table 5. The temperature of glass transition was strongly depended on the moisture content, which is in agreement with the classic theory. At the same time, the seaweeds, which were blanched or washed in a fresh water, did not show any second order transition in the temperature range between -150.0 and + 150.0 °C. It is possible that water-soluble compounds formed maximal freeze concentration, which undergo second order phase transitions. This hypothesis was approved by DSC investigation of surface' mucus of the seaweeds. The solid fraction of the mucus reached 6.8 (0.2) % w.b. and glass

transition phenomena was found.

Table 5. Thermo-physical properties of brown seaweeds with respect to moisture content

Moisture content	Melting energy	$T_{g,on}$	$T_{g,i}$	$T_{g,end}$	T'm	T_{f1}	T_{f2}	Unfreez Water	Ice	Max.Freeze. Solution
% w.b.	J/g	°C						% w.b.		
0.34	n.d.	119.00	120.35	121.87	n.d.	n.d.	n.d.		n.d.	
14.40	n.d.	-41.64	-31.16	-15.56	n.d.	n.d.	n.d.		n.d.	
25.01	n.d.	-87.09	-78.14	-64.63	n.d.	n.d.	n.d.		n.d.	
35.34	21.59	-93.78	-84.35	-75.55	-55.0	-45.3	-28.8	27.43	7.91	70.21
47.91	91.82	-90.25	-86.30	-76.86		-45.1	-17.6	16.81	31.10	75.61
85.66	258.40	-91.28	-84.20	-80.91	-40.0	-17.0	-1.00	6.11	79.55	70.12
94.81	310.30	n.d.	n.d	n.d.	-17.0	n.d.	0.00	1.77	93.04	74.54*
92.97	278.00	cold crystallization detected			-40.0	-16.3	-1.00	9.62		83.35**
* Blannched seaweeds; ** Surface mositure										

Significant difference in incipient melting points were found for raw and washed (blanched) seaweeds. The raw seaweeds showed T'_m at -40.0 °C (high moisture content). While, incipient point of melting for blanched and washed seaweeds was in the range between -20.0 and -17.0 °C. It looks like, that even short-time pre-treatment of seaweeds totally removes some fractions of compounds or make their concentrations insignificantly low.

It was interesting to note, that raw seaweeds with low moisture content showed two melting peaks (T_{f1} and T_{f2}). Which can be explained by salt solution and moisturized carbohydrates.

The state diagram for seaweed

A typical state diagram consists of two curves: the freezing curve and the glass transition curve. The freezing curve represents the influence of the solid matter content on reducing the freezing point. In this investigation it was obtained from the data of the freezing point of seaweed samples with different moisture contents. The decreasing of the freezing point was modeled with the Clausius-Clapeyron equation modified for food by Chen (Chen, 1986)

$$\delta = -\frac{\beta}{M_w}\ln\left(\frac{1-x_s-Bx_s}{1-x_s-Bx_s+Rx_s}\right) \quad (6)$$

The glass transition curve shows the influence of solid content on glass transition temperature. and it was modeled with the Gordon-Taylor equation (Gordon & Taylor, 1952):

$$T_{gi} = \frac{x_s T_{gi.s} + k x_w T_{gi.w}}{x_s + k x_w} \quad (7)$$

The inflection glass transition points values were used for modeling. The parameters of the models are introduced in Table 6 and Figure 3.

Table 6. Parameters and coefficients of the regression models

Equation	Statistical analysis			Coefficients	
	F-ratio	Prob(F)	R^2	R	B
Clausius-Clapeyron	241.57	0	0.96	$1.14*10^{-4}$	$18.67*10^{-2}$
Grodon-Taylor	185.27	0	0.94	k	$T_{gi.s}$
				9.58	151.76

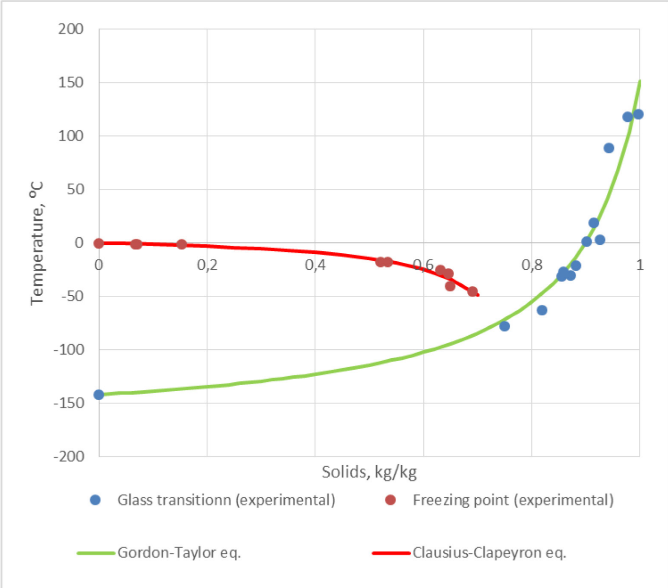

Figure 3. Parameters and coefficients of the regression models.

The application of freeze-drying looks very promising will give an opportunity to detect amount of ice at different processing temperatures. Understand the desirable low temperature condition to avoid glass transition and provide effective drying regimes.

CONCLUSIONS

The drying kinetics of brown seaweed Sacharina Latissima with and without blanching were investigated in temperature range between 10.0 and 38.0 °C. The drying process is more rapid for raw seaweeds. The effective moisture diffusivity coefficient (with respect to shrinkage) was found in the range between 0.5 and 5.0 10-10 m-2/s. Drying temperature influence on lightness and hue color parameter of the seaweeds. The salt content in raw seaweeds influence on the shape of the sorption isotherm at high relative humidifies. The differential scanning calorimetry of raw seaweed samples revealed glass transition at ultra-low temperature range, while it was not found for blanched seaweeds. State diagram for drying and freezing of raw seaweeds was made.

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