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Effect of Different Drying Temperatures on the Moisture and Phytochemical Constituents of Edible Irish Brown Seaweed

Shilpi Gupta Technological University Dublin, shilpi.19may@gmail.com

Sabrina Cox Technological University Dublin, sabrina.cox@tudublin.ie

Nissreen Abu-Ghannam Technological University Dublin, nissreen.abughannam@tudublin.ie

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Effect of different drying temperatures on the moisture and phytochemical constituents of edible Irish brown seaweed

Shilpi Gupta, Sabrina Cox, Nissreen Abu-Ghannam*

School of Food Science and Environmental Health, College of Sciences and Health, Dublin Institute of Technology, Cathal Brugha St., Dublin 1, Ireland

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ABSTRACT

The effect of different temperatures on the drying kinetics and the phytochemical constituents of edible Irish brown seaweed, Himanthalia elongata were studied. This kinetic study involved the modelling of the terms of Fick's diffusion equation, for estimation of the diffusion coefficients. The diffusivity coefficient increased from 5.6×10^{-07} to 12.2×10^{-07} m²/s as the drying temperatures increased with an estimated activation energy of 37.2 kJ/mol. The experimental data was also fitted to different empirical kinetic models, Newton, Logarithmic and Henderson-Pabis, and the goodness of fit for the different models was evaluated. The effect of drying temperatures on the phytochemical constituents in seaweed was also evaluated. Drying at 25 °C resulted in 49% and 51% reduction in the total phenol and total flavonoid content, respectively, as compared to fresh seaweed. However, the reduction declined as the drying temperatures were increased. The scavenging effect on DPPH radical was also greater for the fresh seaweed as compared to the dried form. An increase in the phytochemical content was seen for higher temperatures (35 °C and 40 °C) when the moisture content reduced by 50% indicating that this semi-dry state is even more nutritious than the fresh form and could be an interesting starting point for seaweed processing.

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1. Introduction

Seaweeds are a part of staple diet in the orient as they are nutritionally rich materials [\(Dawczynski, Schubert, & Jahreis, 2007\)](#page-6-0); but to a much lesser extent in the rest of the world. Beneficial nutrients in seaweeds include vitamins, trace minerals, lipids, amino acids, and antioxidants, all of which form the part of a healthy diet ([Athukorala, Kim, & Jeon, 2006](#page-6-0)). Numerous studies have reported on the excellent antioxidant capabilities of seaweeds or their extracts ([Chandini, Ganesan, & Bhaskar, 2008; Cox, Abu-Ghannam, & Gupta,](#page-6-0) [2009\)](#page-6-0). They live in a harsh environment where they are exposed to a wide range of environmental stress such as light, rapid fluctuations in temperatures, osmotic stress and desiccation. These factors can lead to the formation of free radicals and other strong oxidising agents but seaweeds seldom suffer any serious photodynamic damage. This fact implies that seaweed cells have some protective mechanisms and compounds [\(Matsukawa et al., 1997](#page-7-0)).

Being marine in nature seaweeds contain a large amount of water. When fresh, they have $75-85%$ water and $15-25%$ organic components and minerals. Since seaweeds are perishable in their

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fresh state and could deteriorate within a few days after harvest, drying is an essential step before they can be used in industrial processing. Drying decreases the water activity which ultimately retards the microbial growth, helps to conserve the desirable qualities and reduces the storage volume. However, enzymatic and/or non-enzymatic processes that may occur during drying of the fresh plant tissues may lead to significant changes in the composition of phytochemicals ([Capecka, Mareczeek, & Leja, 2005\)](#page-6-0). Studies by [Nicoli, Anese, and Parpinel \(1999\)](#page-7-0) showed that the overall antioxidant capacity of certain foods may be enhanced due to improvement in the antioxidant properties of naturally occurring antioxidants and the formation of Maillard reaction products (MRPs).

Seaweeds are generally sun dried by spreading them over a net, a tarpaulin or over coconut leaves on the ground. In Ireland, drying of seaweeds for the production of different grades of seaweed meal is carried out in rotary dryers heated by coal slack fired kilns [\(http://](#page-6-0) [www.cleanerproduction.ie](#page-6-0)). Different drying methods have been found to greatly affect the nutritional composition of the brown seaweed, Sargassum hemiphyllum [\(Chan, Cheung, & Ang, 1997\)](#page-6-0). [Wong and Cheung \(2001\)](#page-7-0) reported that oven-drying was better than freeze-drying for the extractability and quality of proteins isolated from three subtropical brown seaweeds.

Presently seaweeds are sold in health shops and oriental grocery houses in the dried form. The dried seaweeds can be used as a part

Corresponding author. Tel.: $+353$ 1 402 7570; fax: $+353$ 1 878 8978.

E-mail addresses: [nissreen.abughannam@dit.ie,](mailto:nissreen.abughannam@dit.ie) shilpi.19may@gmail.com [\(](mailto:shilpi.19may@gmail.com)N. Abu-Ghannam).

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of a raw vegetable salad, as a natural seasoning or as a snack with fresh juice. Some studies are available in literature which study the effect of drying on the nutritional properties of seaweeds but no literature is available studying the effect of drying on the phytochemical constituents such as phenols and flavonoids.

The moisture removal and its dependence on the process variables are expressed in terms of the drying kinetics, being essential for the development of a reliable process model. Empirical equations frequently used to model the drying kinetics of food include: Newton, Page, Henderson-Pabis, Logarithmic, Diffusion approach and others [\(Simal, Femenía, Garau, & Roselló, 2005; Vega, Uribe,](#page-7-0) [Lemus, & Miranda, 2007](#page-7-0)).

The present work aimed to study the drying kinetics of H. elongata at a range of temperatures (25, 30, 35 and 40 \degree C) which are applied in the seaweed industry and to evaluate till what extent drying conditions influence the phytochemical content of the seaweeds. These objectives are justified having in mind that the literature lacks some information on the air-drying kinetics of seaweeds either in terms of empirical models or in terms of diffusivity models. Besides, the change in the phytochemical content due to drying has not been explored.

2. Materials and methods

2.1. Seaweed material

Brown seaweed H. elongata was supplied from Quality Sea Veg., Co. Donegal, Ireland. Samples were collected in January 2010, washed thoroughly with freshwater to remove epiphytes and salt and stored at -18 °C until further analysis.

2.2. Drying procedure

Fresh seaweeds were washed and cut manually with stainless steel knife into rectangular samples of approximately $3 \text{ cm} \times$ 0.5 cm \times 0.2 cm. Sample (5 g) was weighed and placed on a flat tray and dried in a hot air oven (Innova 42, Mason Technology, Ireland) at different temperatures of 25, 30, 35 and 40 $^{\circ}$ C. The air velocity was set at 2.0 ± 0.1 m/s as measured with digital anemometer (VWR, Ireland). Samples were withdrawn after every hour until 8 h and then after every 8 h for 24 h. The dry solids content was determined by employing control samples using an oven at 105° C until constant weight of the sample was attained. The relative humidity was monitored with a data logger Grant 1001.

2.3. Drying kinetics expressed in terms of empirical models

The data obtained experimentally for the four different temperatures studied (25, 30, 35 and 40 \degree C) was plotted as a dimensionless variable moisture ratio (MR) versus time:

$$
MR = \frac{W - W_e}{W_0 - W_e} \tag{1}
$$

where W is the moisture content at any time t , W_e the equilibrium moisture content and W_0 is the initial moisture content and all expressed as g water/g dry solids. The experimental data (MR Vs time, t) were fitted to the three different empirical models (Table 1) using STATGRAPHICS Centurion XV (StatPoint Technologies, Inc., Warrenton, VA).

2.4. Estimation of diffusion coefficient

The most widely studied theoretical model in thin layer drying of foods is given by the solution of Fick's second law which was used to fit the experimental drying data. For sufficiently long drying times, the Fick's equation [\(Coulson, Richardson, Backhurst, &](#page-6-0) [Harker, 1987](#page-6-0)) can be simplified to Eq. (2):

$$
MR = \frac{8}{\pi^2} \left(e^{-D_{\text{eff}} t \left(\frac{\pi}{2l} \right)^2} \right) \tag{2}
$$

The above equation assumes that the effective diffusivity (D_{eff}) is constant and that shrinkage of the sample is negligible. The above equation can be further simplified into a straight line:

$$
\ln(MR) = \ln\frac{8}{\pi^2} - D_{\text{eff}}\left(\frac{\pi}{2l}\right)^2 t \tag{3}
$$

Slope of the above line will give the value of effective diffusivity at different temperatures as:

Slope =
$$
-D_{\text{eff}} \left(\frac{\pi^2}{4l^2} \right)
$$
 (4)

The effective diffusivity varies with the temperature according to Arrhenius dependence as:

$$
D_{\rm eff} = D_0 \exp\left(-\frac{E_a}{RT}\right) \tag{5}
$$

where D_0 is diffusivity at an infinite temperature (m²/s), E_a is the activation energy for moisture diffusion (kJ/mol), T is the drying temperature (Kelvin) and R is the gas constant $(8.314$ J/molK).

Upon linearization, the slope indicates the activation energy:

$$
lnD_{\text{eff}} = ln(D_0) + \left(-\frac{E_a}{R}\right)\frac{1}{T}
$$
\n(6)

2.5. Effect of drying on the phytochemical analysis

2.5.1. Preparation of seaweed extracts

The extraction of phenolic compounds from H. elongata was carried out with 60% methanol under nitrogen atmosphere as reported in our previous studies ([Gupta, Rajauria, & Abu-Ghannam,](#page-6-0) [2010\)](#page-6-0).

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2.5.2. Phytochemical and antioxidant analysis

2.5.2.1. Total phenolic content (TPC). The TPC in the extract was determined using Folin-Ciocalteau's phenol reagent ([Taga, Miller,](#page-7-0) [& Pratt, 1984](#page-7-0)). The TPC was expressed as mg gallic acid equivalents (GAE)/100 g dry basis. Fresh weights of each sample were converted into dry weights on the basis of the moisture content.

2.5.2.2. Total flavonoid content (TFC). The TFC was determined by a colourimetric method described by [Liu et al. \(2009\).](#page-6-0) TFC was expressed as mg quercetin equivalents (QE)/100 g dry basis.

2.5.2.3. DPPH radical scavenging assay. This assay was carried out as described by [Yen and Chen \(1995\)](#page-7-0), with some modifications. Analysis were performed in a 96-well microplate with 1:1 ratio of 100 μ l of DPPH solution (165 μ M) and 100 μ l of sample. The reaction mixtures were incubated for 30 min at 25 \degree C in dark and absorbance was measured at 517 nm in a microplate reader (Powerwave, Biotek, VT, USA). The ability to scavenge the DPPH radical was calculated as:

Scavenging capacity(
$$
\mathscr{X}
$$
) = $\left[1 - \left(\frac{A_{sample} - A_{sampleblank}}{A_{control}}\right)\right] \times 100$ (7)

where, A_{control} is the absorbance of DPPH solution without sample, Asample is the absorbance of DPPH solution plus test sample and Asample blank is the absorbance of the sample without any DPPH solution. Calculated EC_{50} values indicate the concentration of sample required to scavenge 50% DPPH radicals. The lower the EC_{50} value of the sample, the higher is the antioxidant capacity.

2.6. Statistical analysis

All the experiments were carried out in triplicate and replicated at least twice. The goodness of fit of the tested mathematical models to the experimental data was evaluated from the coefficient of determination (R^2), Sum square error (SSE; Eq. (8), root mean square error (RMSE; Eq. (9) and the chi-square (χ^2 ; Eq. (10) between the predicted and experimental values.

$$
SSE = \frac{1}{N} \sum_{i=1}^{N} \left(MR_{exp} - MR_{pred} \right)^2
$$
 (8)

$$
RMSE = \sqrt{\frac{1}{N} \sum_{i=1}^{N} (MR_{exp} - MR_{pred})^2}
$$
(9)

$$
N^{2} = \frac{\sum_{i=1}^{N} (MR_{\exp,i} - MR_{\text{pred},i})^{2}}{N - Z}
$$
 (10)

where $MR_{exp,i}$ and $MR_{pred,i}$ are the experimental and predicted moisture ratio, N is the number of observations and z is the number of constants.

Fig. 1. Experimental drying curves of H. elongata at different temperatures (\diamond : 25 °C; \Box : 30 °C; \triangle : 35 °C; \bigcirc : 40 °C).

Table 2

3. Results and discussion

3.1. Drying curves

Moisture content of the fresh H. elongata was approximately 4.05 ± 0.05 kg water/kg dry matter. Fig. 1 shows the variation of moisture content as a function of time at the four temperatures studied. All the drying curves show a clear exponential tendency and an increase in the temperature accelerated the drying process. There was significant difference in the moisture content with different drying temperatures ($P < 0.05$). At 25 °C the drying rate was minimal and approached equilibrium after 8 h whereas equilibrium at 40 \degree C was attained after 5 h, representing 37.5% reduction in the total drying time. In addition, as the temperature increased, there was an increase in the rate of mass transfer (water) to achieve similar equilibrium moisture content (approximately 0.98 g water/100 g d.m) ([Miranda, Maureira, Rodríguez, & Vega-](#page-7-0)[Gálvez, 2009](#page-7-0)). Seaweeds have a high rate of moisture loss when kept in air and thus have a tendency to loose water quickly. Generally, the seaweed industry employs outdoor drying under atmospheric conditions. Thus, the drying temperatures applied in the present study were low to imitate the air-drying conditions in the industry. Drying was carried out under controlled conditions to achieve optimum drying time which will be short and will not reduce the final quality of the dried product. Moreover, preliminary experiments had shown that drying the seaweeds at temperatures above 50 \degree C resulting in colour darkening within 2 h with a complete loss in the antioxidant properties. The percentage reduction of antioxidants for the samples dried at 50 \degree C was 87%.

3.2. Estimation of diffusion coefficient

The traditional method for studying the mass transfer at a nonstationary state for the drying of foodstuffs is the Fick's equation (Eq. [\(2\)](#page-2-0)), from which the effective diffusivity coefficient (D_{eff}) is determined. Effective diffusivities of dried seaweeds at different temperatures were obtained from the gradient of the plots of ln (MR) versus drying time (t) (Eq. [\(3\)\)](#page-2-0) for 25, 30, 35 and 40 °C with slopes of 0.3483 h⁻¹, 0.4841 h⁻¹, 0.5224 h⁻¹ and 0.6566 h⁻¹, respectively. Table 2 shows the results of the fitting to Eq. [\(3\),](#page-2-0) which allowed the calculation of the diffusion coefficients, D_{eff} , at the different temperatures by Eq. [\(4\)](#page-2-0). The diffusivity increased from $5.6 \times 10^{-07} \text{ m}^2/\text{s}$ to $12.2 \times 10^{-07} \text{ m}^2/\text{s}$ as the temperature was

Fig. 2. Variation of effective diffusivity (m^2/s) with inverse of temperature.

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Table 3

Statistical indices upon modelling the drying of H. elongata at a range of temperatures.

increased. Similar behaviour of D_{eff} has been reported for okra ([Doymaz, 2005](#page-6-0)), aloe vera [\(Vega et al., 2007\)](#page-7-0) and onions [\(Mota,](#page-7-0) [Luciano, Dias, Barroca, & Guiné, 2010\)](#page-7-0). However, the diffusivity values obtained in the present study are higher than those reported in literature for other vegetables ([Chong et al., 2008; Vega et al.,](#page-6-0) [2007\)](#page-6-0). The reason for this could be the higher initial water content of H. elongata allowing greater diffusion coefficients, since the process of diffusion is favored in products with higher proportions of water and lower proportions of solids ([Guiné &](#page-6-0) [Fernandes, 2006\)](#page-6-0). Diffusivity values were then used to fit Eq. [\(5\),](#page-2-0) to estimate the values of the diffusivity for an infinite temperature, D_0 , and the activation energy for moisture diffusion, E_a . The results show a high quality fitting with a R^2 value of 0.9484 ([Fig. 2](#page-3-0)). The value obtained for the diffusion coefficient at an infinite temperature, $D_{\rm o}$, was 1.87 m 2 /s, with activation energy for moisture diffusion, E_a , to be 37.2 kJ/mol. The values of activation energy obtained are in line with those reported for other vegetables [\(Doymaz, 2005;](#page-6-0) [Mota et al., 2010; Vega et al., 2007\)](#page-6-0).

3.3. Modelling of drying curves

Although the drying kinetics was temperature-dependent, the differences in the moisture content decreased as the system reached equilibrium. The drying kinetics data obtained for the four temperatures was fitted to three empirical kinetic models ([Table 1\)](#page-2-0). It was observed that R^2 values (Table 3) ranged from 0.948 to 0.995 for the different models. The fact that drying kinetics was temperature-dependent could be ascertained from the fact that the value of the parameter k' (Table 4) increased for all the models as the drying temperature was increased. Fig. 3 shows the predicted and experimental points obtained for the four temperatures with the different models tested: Newton, Henderson-Pabis and Logarithmic. From the results obtained it can be verified that the three models used in this study show a good predicting capacity, and revealed good performance for the temperatures tested, over the entire duration of the drying process. However, Newton model had

Fig. 3. Experimental and predicted drying curves for (a) Newton; (b) Logarithmic and (c) Henderson–Pabis model for the four temperatures. (\diamond : 25 °C; \Box : 30 °C; \triangle : 35 °C; \bigcirc : 40 $\,^{\circ}$ C).

Table 4

Results of fitting of drying kinetics to the three models (values in curved brackets are the standard error and values in square brackets are 95% confidence intervals).

 k , a and c are the model parameters.

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Fig. 4. Graphic representation of the influence of temperature on parameter (k) from different models. (O: Henderson-Pabis ($R^2 = 0.9598$); \Box : Logarithmic ($R^2 = 0.9596$); \diamond : Newton ($R^2 = 0.9632$)).

the lowest R^2 values among all the three models. The values of the standard error of the parameter 'k' in the Newton model vary between 3% and 12% for all the temperatures studied. The values of standard error for Henderson-Pabis model were within acceptable range for parameter 'a' $(2\%–6\%)$ and 'k' (5%–13%). Regarding the Logarithmic model, the values for the standard errors of parameter 'a' vary from 3% to 8% and 'k' varies from 6% to 19% are within the acceptable range, but the standard errors of the parameters 'c' for all the temperatures is of the same order of the value itself or greater.

In order to prove the dependence of parameter 'k' on the drying temperature, the Arrhenius equation was applied, graphically representing ln k versus 1/T ([Simal et al., 2005](#page-7-0)). Straight lines were obtained with regression coefficients (R^2) higher than 0.98 (Fig. 4), from whose slopes activation energies of 46.7, 47.4, and 41.8 kJ/mol were obtained for the parameters of Newton, Logarithmic and Henderson-Pabis model, respectively. Based on the similarities between the activation energy of the diffusivity coefficient (37.16 kJ/mol) and the parameter 'k' as obtained above for different models, in addition to the values reported by [Senadeera, Bhandari,](#page-7-0) [Young, and Wijesinghe \(2003\)](#page-7-0) for other vegetables (12.87–58.15 kJ/ mol), the parameter 'k' can be considered as pseudodiffusivity. The parameter 'k' represents a pseudodiffusional behaviour of matter transfer as stated in Fick's second law. The ANOVA carried out on the parameters 'a' of Henderson-Pabis model showed no statistically significant difference ($P > 0.05$) of these parameters as related to temperature, suggesting they probably depend more on the characteristics of the tissue and/or the drying air flow. [Vega et al.](#page-7-0) [\(2007\)](#page-7-0) also reported similar observations for the parameter 'a' of HP model.

3.4. Effect of drying on the phytochemical constituents

In our previous studies we had reported that methanolic extracts from H. elongata have high antioxidant activity [\(Cox et al.,](#page-6-0) [2009\)](#page-6-0). Processing of any kind will affect content, activity and bioavailability of bioactive compounds. The TPC was monitored for H. elongata dried at different temperatures over the entire duration of drying (Fig. 5a). The initial content of total phenol was 1.55 ± 0.026 g GAE/100 g dry seaweed. The content of total phenol was found to be higher than those reported for other common algae such as Laminaria, Undaria, Scytosiphon, Tunbinaria ([Chandini et al.,](#page-6-0) [2008; Jiménez-Escrig, Jiménez-Jiménez, Pulido, & Saura-Calixto,](#page-6-0) [2001; Kuda, Tsunekawa, Hishi, & Araki, 2005](#page-6-0)). Overall drying at different temperatures resulted in a reduction in the TPC; however the content was still higher than the values reported for dried Scytosiphon lomentaria ([Kuda et al., 2005\)](#page-6-0). Drying at lower temperatures (25 \degree C and 30 \degree C) resulted in a continuous reduction of TPC (Fig. 5a) although a small increase was seen (at 4 h) when the moisture content had reduced by half. But these values were a lot less than that in the fresh seaweed. For higher temperatures (35 \degree C and 40 \degree C) an increase in the TPC content was seen for the first 2 h after which it started decreasing (Fig. 5a). Maximum increase of 41% in the TPC was seen after drying at 40 \degree C for 2 h. Since identical

Fig. 5. Effect of different drying temperatures on the (a) total phenolic content and (b) total flavonoid content of H. elongata. Results are expressed as per 100 g dry basis. (\diamond : 25 °C; \Box : 30 °C; \triangle : 35 °C; \triangle : 40 °C).

amounts of sample were taken for fresh and dried seaweeds, there was no influence of residual moisture on the antioxidant capacity or TPC of the samples. This increase could be related to the developmental changes and wound-like response due to drying. Dixon and Paiva (1995) reported that plants respond to wounding with increase in phenolic compounds involved in the repair of wound damage. However, at the end of the 24 h drying period a significant reduction (29 -51%) in the TPC was seen for H. elongata dried at different temperatures ($P < 0.5$). Maximum reduction of 51% in the TPC was seen in H. elongata dried at 25° C whereas a reduction of only 29% was seen when drying was done at 40 \degree C as compared to fresh seaweed. A probable reason for this could be the long drying time of seaweeds at 25 \degree C to achieve a similar equilibrium moisture content as compared to when drying at 40° C. Jiménez-Escrig et al. (2001) reported a 98% reduction in the TPC content on brown seaweed Fucus dried at 50 \degree C for 48 h. Garau et al. (2007) reported that longer drying times resulted in a reduction of TPC for orange by-products. Also, the lower drying temperatures used in the present study probably did not inactivate the oxidative enzymes completely, which may have in turn resulted in some oxidation of the phenolic substances and resulted in a relatively lower phenolic content. Decrease in TPC during drying can also be attributed to the binding of polyphenols with other compounds (proteins) or the alterations in the chemical structure of polyphenols which cannot be extracted or determined by available methods (Martín-Cabrejas et al., 2009; Qu, Pan, & Ma, 2010).

[Fig. 5](#page-5-0)b shows the variation in the TFC for the four different temperatures studied. The TFC in the fresh seaweeds was 0.49 g \pm 0.019 QE/100 g dry seaweed. TPC reduced continuously for the lower temperatures but at higher temperatures it increased initially and then decreased. Drying led to a reduction in the TFC as well, although the % reduction declined as the drying temperature increased. A percentage reduction of 49% and 30% was seen at 25 \degree C and 40 \degree C, respectively.

The antioxidant capacity of fresh and dried H. elongata was determined by the DPPH radical scavenging assay. Minimum EC_{50} value was seen for fresh seaweeds $(10 \mu g/ml)$. Reduction in TPC values at various drying temperatures was accompanied by a reduction in the antioxidant potential as well. Drying resulted in significant decrease ($P < 0.05$) in the antioxidant activity exhibited by the reduction in DPPH free radical scavenging activity, i.e. higher EC₅₀ of 25 (25 °C and 30 °C) and 50 μ g/ml (35 °C and 40 °C) as compared to an EC_{50} of 10 μ g/ml for fresh samples.

Li, Smith, and Hossain (2006) had reported that a combination of high drying temperatures and long drying times might destroy some of the phenol compounds. In addition, all the plant cell components adhere together in the absence of water, and possibly making the extraction with solvent more difficult; as a result, the overall recoveries might be lower than expected (Li et al., 2006). Sun drying and subsequent storage of algae have been reported to cause a reduction in the levels of labile antioxidants such as L-ascorbate and GSH (Burritt, Larkindale, & Hurd, 2002; Jiménez-Escrig et al., 2001). The drying process would generally result in a depletion of naturally occurring antioxidants in raw materials from plants. Intense and/or prolonged thermal treatment may be responsible for a significant loss of natural antioxidants, as most of these compounds are relatively unstable (Lim & Murtijaya, 2007).

4. Conclusion

This study showed that the drying kinetics of seaweeds can be accurately predicted using the empirical models of Newton, Logarithmic or Henderson-Pabis model. The moisture transfer can be described by diffusion and the temperature dependence of the effective moisture diffusivities was shown to follow an Arrhenius relationship. Drying reduced the phytochemical constituents in the seaweed. A reduction of 29% in the TPC and 30% in the TFC was seen when H. elongata was dried at 40° C. However, an important increase of 41% in the TPC was nonetheless observed when the seaweed was dried up to 50% moisture content. This would mean that the semi-dried form of seaweeds which is even more nutritious than the raw state could be used for the development of health promoting seaweed based products. However, the results also showed that processing of H. elongata by drying resulted in a substantial reduction of the phytochemicals which leads to the fact that new research into protecting antioxidant properties of seaweeds upon processing would be needed.

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