

Effects of temperature on drying kinetics and biochemical composition of *Caulerpa lentillifera*

¹Fakhrulddin, I.M., ²Ramaiya, S.D., ^{1,3}Muta Harah, Z., ^{1,4}Nur Leena Wong, W.S.,
^{5,*}Awang, M.A. and ⁶Ismail, N.I.M.

¹International Institute of Aquaculture and Aquatic Sciences (I-AQUAS), Universiti Putra Malaysia, 71050 Port Dickson, Negeri Sembilan, Malaysia

²Department of Crop Science, Faculty of Agriculture Sciences and Forestry, Universiti Putra Malaysia Bintulu Campus, 97008 Bintulu Sarawak, Malaysia

^{3,4}Department of Aquaculture, Faculty of Agriculture, Universiti Putra Malaysia, 43400 UPM Serdang, Selangor Darul Ehsan, Malaysia

⁵Faculty of Food Science and Nutrition, Universiti Malaysia Sabah, Jalan UMS, 88400 Kota Kinabalu Sabah, Malaysia

⁶Department of Science and Mathematics, Centre for Diploma Studies, Universiti Tun Hussein Onn Malaysia, Pagoh Higher Education Hub, 84600, Pagoh, Johor, Malaysia

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Abstract

Caulerpa lentillifera (phylum: Chlorophyta) is a seaweed that is widely consumed by local communities across Asia. The effects of different oven-dried temperatures on the drying kinetics and phytochemical constituents of *C. lentillifera* were studied. This kinetic study used three different thin-layer models, i.e., Lewis, Henderson and Pabis, and Logarithmic models. Among these, the Logarithmic model was the most suitable model that could be determined the seaweed drying behaviours (R-square > 0.9923, RMSE < 0.0004). Drying the *C. lentillifera* at 50°C resulted in a higher extraction yield (1.00%) with a significant concentration of total phenolic content and total flavonoid content at 1.027 mg GAE/g, and 59.655 mg RU/g, respectively.

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1. Introduction

Marine macroalgae (seaweed) is widely regarded as nutritious food and has been found to have extensive applications in many industries, including food, fitness, and beauty. They were divided into three categories based on their pigment colours, i.e., Rhodophyta (e.g., *Gracilaria edulis*), Phaeophyta (e.g., *Sargassum horneri*), and Chlorophyta (e.g., *Caulerpa lentillifera*), which encompass at least 600, 200, and 1200 species, respectively (Venugopal, 2011). They are known to exhibit numerous biological activities, such as antioxidant, antibacterial, anticancer, anticoagulant, and antiviral (Vairappan *et al.*, 2001; Athukorala *et al.*, 2007). Aside from being low-calorie, seaweeds contain high vitamins, proteins, minerals, polyphenols, polysaccharides, and dietary fibres (Burtin, 2003; MacArtain *et al.*, 2007). Therefore, they act as potential functional food capable of improving human health and preventing diseases, such as diabetes, hypertension, and

cardiovascular diseases (Menrad, 2003). Seaweed is also commonly used as chemical additives and thickening agents, including carrageenan, agar, and alginate (Holdt and Kraan, 2011).

Caulerpa lentillifera (phylum: Chlorophyta), also known as green caviar or sea grapes is widely consumed in fresh or salad form by the local communities in parts of Asia, particularly in the coastal region (Gan *et al.*, 2011; Zawawi *et al.*, 2015). The seaweed is green in colour and grass-like, with a delicate and succulent texture (Amata *et al.*, 2018). The demand for *C. lentillifera* has increased over the year due to its high nutritive values and wide applications as cosmetic and animal feed. Nonetheless, the short storage period of raw *C. lentillifera* causes a supply problem in a region far away from the coast. Hence, the seaweed is normally preserved through dehydration to prolong the storage period and resolve the transportation issue, as fresh *C. lentillifera* contains high humidity (up to 80%) (Gupta *et*

*Corresponding author.

Email: ma.awang@ums.edu.my

al., 2011).

The drying method is proven to be the most effective method in preserving seaweed to avoid progressive degradation and biochemical changes (Nurjanah et al., 2016). Various food drying techniques have been developed, including sun-, oven-, vacuum-, and microwave-drying (Zhang et al., 2010). The main objective of drying is to inhibit microbial activity on food through the removal of water (dehydration), therefore slowing down chemical degradation in food (Gupta et al., 2011). A common industrial practice for preserving seaweed is through drying at 50–80°C (Djaeni and Sari, 2015). Notwithstanding, the drying process consumes high energy as well as induces physical and chemical changes (texture, colour, and chemical composition) (Guine and Barroca, 2012), ultimately affecting the consistency, colour, scent, nutrition, and phytochemistry of the seaweed (Chan et al., 1997). In this regard, it is important to design and determine the optimum drying temperature for *C. lentillifera* to minimise energy consumption and biochemical alteration in seaweed during the drying process (Stramarkou et al., 2017). Herein, this study aimed to identify the optimum drying temperature from the calculation using several mathematical modelling. The crude extraction yield, total phenolic, and total flavonoid content of the dried seaweed were also investigated.

2. Materials and methods

2.1 Drying experiment

Caulerpa lentillifera seaweed was collected from the Blue Lagoon, Port Dickson, Negeri Sembilan (GPS coordinate: 2°24'55.9"N 101°51'15.7"E). The sample was washed using running water to remove the sand particles and other residues. Then, 100 g of fresh seaweeds were placed in a tray (35×50 cm) and dried at various temperatures between 40–80°C (Awang et al., 2021), using an oven (Model 30-750, Memmert, Germany) equipped with a temperature controller and a suction fan. The weight change was measured at 10 mins intervals using an electronic balance (Pioneer, Shimadzu, Japan). The drying, cooling, and weighing processes were repeated until a constant weight was obtained. The moisture content of the seaweed was measured using a Halogen Moisture Analyzer (Model HB43, Mettler Toledo, USA). All the experiments were conducted in

triplicate.

2.2 Extraction

The dried seaweed was ground into powder using a blender (Model EBM-9182, Elba, Malaysia). Approximately 10 g of dried seaweed was extracted with 200 mL of ethanol (70%) at 60°C for 1 hr (Awang et al., 2017). The extract was filtered using a vacuum pump to remove the solid residues, followed by concentrating the crude extract using a rotary evaporator and then drying in an oven at 40°C for further analysis.

2.3 Determination of drying kinetics

The experimental data obtained from drying experiments were fitted into three thin-layer models (Lewis, Henderson and Pabis, logarithmic model) in Table 1 to calculate the drying kinetic pattern. All experimental data were expressed in the dimensionless moisture ratio (MR) as shown in Equation (1). The drying process was assumed to be controlled by the external resistance between the samples and surrounding drying air in the oven.

$$MR = \frac{M - M_e}{M_o - M_e} \quad (1)$$

where M is the moisture content, M_o is the initial moisture content, and M_e is the equilibrium moisture content. The goodness-of-fit for the selected models was evaluated using the determination coefficient (R^2) and root-mean-square error (RMSE) according to Equation (2) and (3), respectively.

$$R^2 = 1 - \left[\frac{\sum_{i=1}^N (MR_{exp,i} - MR_{pre,i})^2}{\sum_{i=1}^N (MR_{exp,i} - MR_{exp,mean})^2} \right] \quad (2)$$

$$RMSE = \left[\frac{1}{N} \sum_{i=1}^N (MR_{exp,i} - MR_{pre,i})^2 \right]^{\frac{1}{2}} \quad (3)$$

2.4 Evaluation of total phenolic content and total flavonoid content

The colourimetric assay of total phenolic content (TPC) was performed using Folin-Ciocalteu reagent according to the method described by Silva et al. (2007) and Ramaiya et al. (2019) with some modifications. Briefly, 1 mL of crude extract (1 mg/mL) was mixed with 2.5 mL of 10% Folin-Ciocalteu reagent, followed by the addition of 2 mL of 7% sodium carbonate after 3

Table 1. Thin-layer models of drying kinetics

Model	Equations	References
Lewis	$MR = \exp(-kt)$	Kaleta and Górnicki (2010)
Herderson and Pabis	$MR = a \exp(-kt)$	Diamante et al. (2010)
Logarithmic	$MR = a \exp(-kt) + b$	Kaur and Singh (2014)

Note: The constant a is the number of experiments, whereas b is the number of independent variables in the regression analysis.

mins. The mixture was incubated in the dark for 90 mins at room temperature and the absorbance of samples was measured at 725 nm using a UV-Vis spectrophotometer (Shimadzu UV-1800, Kyoto, Japan). Gallic acid was used as a standard to construct a calibration curve. The results are expressed as milligrams of gallic acid equivalent (mg GAE) per gram extract (g extract). The total flavonoid content (TFC) of samples was estimated according to the protocol outlined by Al-Matani *et al.* (2015). Briefly, 1 mL of the sample was added with 0.3 mL of 5% sodium nitrite. After 5 mins, 0.3 mL of 10% aluminium chloride was then added, followed by 2 mL of 5% sodium hydroxide. The absorbance of samples was measured at 510 nm using a spectrophotometer (Shimadzu UV-1800, Kyoto, Japan), and Rutin was used as a standard for the construction of the calibration curve. The results were expressed as milligrams of Rutin equivalent (mg RU) per gram extract (g extract). Both results of TPC and TFC were calculated using Equation (4).

$$TPC \frac{mg \text{ GAE}}{g \text{ extract}} \text{ or } TFC \frac{mg \text{ RU}}{g \text{ extract}} = C \times V m \quad (4)$$

Where C = concentration of gallic acid/ RU from the standard curve, V = volume of sample (mL), and m = mass of sample (g).

3. Results and discussion

The initial moisture content of the fresh seaweed was 81% (w/w). The time required to reach the equilibrium moisture content during the drying of seaweed was 210, 150, 100, 80, and 60 mins at 40, 50, 60, 70, and 80°C, respectively (Figure 1). Indicating that the drying time taken for 40°C was the longest, probably due to the heat being applied at that temperature not being sufficient for vaporisation to take place. The negative correlation between the drying temperature and the drying time was in agreement with many previous studies that reported on processed pumpkin slices (Akpınar, 2006; Doymaz, 2007), Asian white radish (Lee and Kim 2009), and *Melastoma malabathricum* (Awang *et al.*, 2021). Table 2 shows the experimental results of the drying process at different temperatures after being analysed using three thin-layer models. It was evident that the experimental data (Figure 1) satisfactorily matched the models based on the high correlation of determination ($R^2 > 0.95$, RMSE < 0.007).

The quality of dried seaweed was also evaluated based on the yield of extraction, TPC, and TFC. Figure 2 shows a significant increase in crude yield of extraction from 40 to 80°C. TPC and TFC, in contrast, decreased significantly beyond 50°C (Figure 3). According to Badmus *et al.* (2019), drying at an elevated temperature reduces the TPC, TFC, and antioxidant activity of five

species of brown seaweeds (*Fucus spiralis*, *Laminaria digitata*, *Fucus serratus*, *Halidrys siliquosa*, *Pelvetia canaliculata*). Similar to terrestrial plants, the extract of *Clinacanthus nutans* (herbal medicinal plant) exhibited the highest TPC and antioxidant content at 55°C, while the bioactive compounds in the extract were degraded at high temperature (Baharuddin *et al.*, 2018). Previous studies conducted by Altemimi *et al.* (2016) showed that the peach extract that had been heated at above 50°C exhibited a significantly lower TPC. Similarly, the TPC of *Schizophyllum commune* only showed an elevation when heated from 30 to 42.5°C, followed by a decrease in TPC from 42.5 to 55°C (Yim *et al.*, 2013). Nonetheless, these observations are not consistent in all species. For example, the study conducted by Badmus *et al.* (2019) showed that the TFC in dried brown seaweed, *Halidrys siliquosa* had no difference from the fresh one, similar to *Laminaria digitata* in terms of its antioxidant activity.

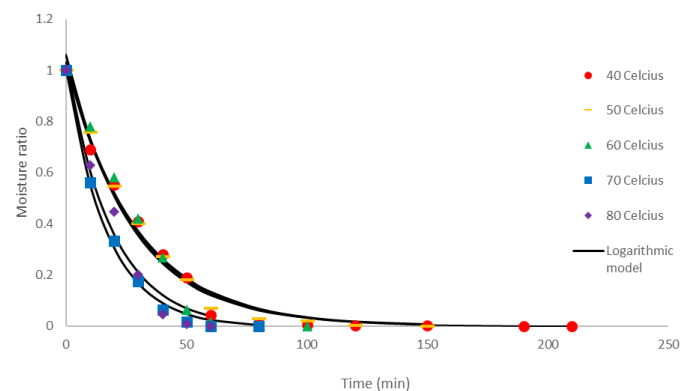


Figure 1. Experimental data and logarithmic model for drying kinetics with different temperatures (°C).

Bener *et al.* (2013) found that an increase in the temperature did not improve the extraction and isolation rate of bioactive compounds but caused them to degrade instead. During seaweed extraction, the TPC decreased after the drying process could be due to the binding of polyphenols with other compounds (e.g., protein) or changes in the chemical structure of polyphenols that resulted in poor extraction using conventional methods (Mrad *et al.*, 2012). A study by Ling *et al.* (2015) reported that the oven-dried (40°C) seaweeds species *Kappaphycus alvarezii* has higher total phenolic, flavonoid, anthocyanin, and carotenoid content, as well as stronger scavenging and reducing abilities than those dried at 80°C. On the other hand, drying seaweeds at a low temperature (20°C) caused the TPC and TFC to drop by 49 and 51%, respectively (Gupta *et al.*, 2011). These variations in phytochemical content and antioxidant activities as a result of different drying temperatures suggested that the drying temperature significantly impacts either the content or extractability of potentially antioxidant components (Cruces *et al.*, 2016). Although we have determined that drying *C. lentillifera* at 50°C

Table 2. Analysis of data using thin-layer model at different temperatures

Model	Temperature (°C)	Constant (k, a, b)	R ²	RMSE
Lewis	40	$k = 0.0339$	0.9868	0.0007
	50	$k = 0.0331$	0.9912	0.0005
	60	$k = 0.0346$	0.9581	0.0029
	70	$k = 0.0593$	0.9957	0.0003
	80	$k = 0.0516$	0.9741	0.0019
Herdenson and Pabis	40	$k = 0.0343$ $a = 1.0116$	0.9870	0.0070
	50	$k = 0.0341$ $a = 1.0302$	0.9922	0.0005
	60	$k = 0.0365$ $a = 1.0604$	0.9625	0.0026
	70	$k = 0.0597$ $a = 1.0080$	0.9957	0.0003
	80	$k = 0.0528$ $a = 1.0278$	0.9751	0.0018
Logarithmic	40	$k = 0.0343$ $a = 1.0112$ $b = 0.0000$	0.9870	0.0006
	50	$k = 0.0341$ $a = 1.0304$ $b = -0.0002$	0.9923	0.0004
	60	$k = 0.0370$ $a = 1.0551$ $b = 0.0060$	0.9734	0.0027
	70	$k = 0.0590$ $a = 1.0121$ $b = -0.0050$	0.9964	0.0002
	80	$k = 0.0520$ $a = 1.0340$ $b = -0.0070$	0.9768	0.0016

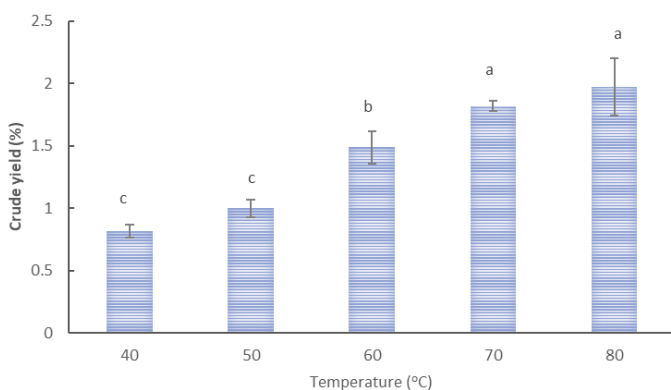


Figure 2. Total crude extraction yield of *C. lentillifera* at different drying temperatures (°C). Bars with different notations are significantly different at $p < 0.05$ (ANOVA, Tukey's test).

has an optimum drying time and may preserve the TPC and TFC, it may not apply to all seaweed species due to the variation in cell walls and physiology of seaweeds (Cox *et al.*, 2012).

4. Conclusion

From the results presented here, the choice of drying temperature can significantly influence the yield and presence of phytochemical compounds in *C. lentillifera*. Higher drying temperatures increase the yield of extract. However, adverse effects on the phytochemical content

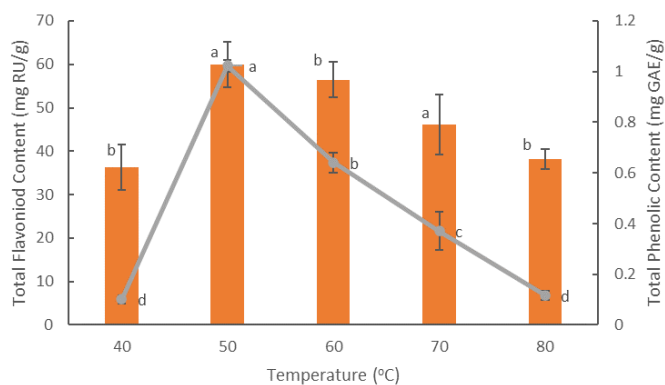


Figure 3. Total phenolic content (bar) and total flavonoid content (line) in *C. lentillifera* of different drying temperatures (°C). Bars and lines with different notations are significantly different at $p < 0.05$ (ANOVA, Tukey's test).

were also observed due to degradation at higher drying temperatures. The logarithmic model was the best fit model to describe the drying process of *C. lentillifera* under the stipulated conditions. The optimal drying temperature of *C. lentillifera* can serve as a guide for the food industry in improving the quality of seaweed-based products.

Conflict of interest

The authors declare no conflict of interest.

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