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## Drying Behavior of Osmotically Air-dried Semi-refined Carrageenan

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**Abstract** The study investigated the effects of osmotic dehydration (OD) as pre-treatment to the drying behavior of mechanically air-dried semi-refined carrageenan (SRC). It aimed to analyze mass transfer during OD as affected by temperature, as well as evaluate the drying properties of the powdered product. As observed, OD was able to reduce product moisture content from 92.23-94.45% to 33.55-47.85% after two hours of processing time. OD plots showed rapid initial moisture loss and solute gain followed by slow rates until equilibrium condition was reached. Higher OD temperature also improved mass transfer, thereby enhancing solute deposition on the samples and moisture migration into the osmotic medium. As a result, OD could potentially cut energy cost of subsequent air-drying because of reduced initial moisture content of the product. However, processing at higher OD temperature can also decrease drying rate and power rating due to excessive sucrose gain.

**Keywords:** osmotic dehydration; semi-refined carrageenan; mass transfer; diffusion; empirical models

### Introduction

Osmotic dehydration is a pre-treatment procedure commonly performed to food products prior to air-drying. With this procedure, the food is immersed in a hypertonic solution (Silva *et al.*, 2012) which has higher osmotic pressure, thereby causing partial removal of water from the food material (Khan, 2012; Souraki, Ghavami and Tondro, 2013). Indeed, this process could be regarded more accurately as a concentration process rather than drying (Brennan, 1994; qtd in Souraki, Ghavami and Tondro, 2013).

Osmotic dehydration is widely considered as an energy-efficient method as time for consequent air-drying is reduced (Ruiz-Lopez *et al.*, 2010; qtd in Herman-Lara *et al.*, 2013). It has been reported that osmotic dehydration is able

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to lessen the weight of fresh vegetables and fruits by up to 50% (Rastogi and Raghavararo, 1997). Added benefits of osmotic dehydration include reduction of damage by heat to the flavor and color of the food by inhibiting browning by enzymes and increase in the glass transition temperature and collapse temperature of the food, leading to a better appearance and porosity (Botha, Oliveria and Ahrne, 2012).

Semi-refined carrageenan is a hydrocolloid produced from alkali treatment of seaweeds. Despite having inferior quality relative to refined carrageenan, it continues to be a major aquaculture product in the Philippines since its production is comparatively easier and more practical for local industries. Though mechanical drying of semi-refined carrageenan has been widely studied, subjecting it to osmotic dehydration has never been fully investigated. Thus, this study was conducted to evaluate the drying properties of semi-refined carrageenan with osmotic dehydration as pre-treatment.

## **Materials and methods**

### ***Extraction of Semi-refined Carrageenan***

Local industry practices were followed in the extraction of semi-refined carrageenan. Dried seaweeds of the specie *Kappaphycus alvarezii* were obtained from local suppliers. Samples were initially rehydrated in distilled water for 30 minutes. Afterwards, they were cooked in an alkali solution with the ratio of 300 g KOH and 22.4 g KCl dissolved in four (4) liters of water for every kilogram of seaweed. Cooking was performed for approximately two (2) hours at 80-85<sup>o</sup>C temperature range.

### ***Osmotic Dehydration***

After cooling at ambient conditions, the cooked seaweeds were washed in distilled water and chopped into uniform sizes of approximately one (1) inch long and 2.50 mm thick. Samples with weights  $5.10 \pm 0.10$  grams were then immersed in prepared saturated sucrose solutions at temperatures of 30<sup>o</sup>, 40<sup>o</sup>, and 50<sup>o</sup>C. Osmotic medium was not stirred during the experimentation; however, ratio of the product and solution in all trials was maintained at 1:20. At this proportion, the concentration is strong enough so that driving force for mass diffusion becomes nearly uniform (Rastogi *et al.*, 2014).

### ***Mass Transfer Modelling***

To create the diffusion models, destructive sampling was employed and samples were submerged in the sucrose solution at 5, 15, 25, 35, 45, 60, 75, 95, and 120 minutes. After which, they were subjected to oven method (using CENCO air oven) to determine their moisture content.

Mass transfer during osmotic dehydration was analyzed using parameters on solid gain (SG) and water loss (WL):

$$WL = \frac{(MC_o)(W_o) - (MC_t)(W_t)}{W_o} \quad \text{equation (1)}$$

$$SG = \frac{S_t - S_o}{W_o} \quad \text{equation (2)}$$

where:  $W_o$  = initial weight of seaweed (g)  
 $W_t$  = weight of seaweed after osmotic dehydration at any time t (g)  
 $MC_o$  = initial moisture content of seaweed (%)  
 $MC_t$  = moisture content after osmotic dehydration at any time t (%)  
 $S_o$  = initial weight of the solids (g) =  $(1 - MC_o)(W_o)$   
 $S_t$  = weight of solids after osmotic dehydration at any time t (g)  
 $= (1 - MC_o)(W_o)$

On the other hand, mass transfer kinetics was evaluated using Page model where the dependent variables are the dimensionless amount of solute gain ( $W_S$ ) and water loss ( $W_A$ ):

$$W_{A \text{ or } S} = \exp(-kt^n)$$

where:  $W_{A \text{ or } S} = \frac{MC_{t, A \text{ or } S} - MC_{eq, A \text{ or } S}}{MC_{O, A \text{ or } S} - MC_{eq, A \text{ or } S}}$   
 $=$  water loss (A) or solute gain (S) ratio  
 $MC_{O, A \text{ or } S}$  = initial amount of water or solute, %  
 $MC_{t, A \text{ or } S}$  = amount of water or solute at time t, %  
 $MC_{eq, A \text{ or } S}$  = equilibrium amount of water loss or solute gain, %  
 $t$  = time  
 $k, n$  = constants

To evaluate the influence of temperature on osmotic dehydration, ANOVA and Pearson product-moment correlation were used.

### ***Air-Drying of Samples***

For product evaluation, the semi-refined carrageenan was subjected to osmotic dehydration for seven (7) minutes. Afterwards, samples were air-dried at 60°C using a laboratory-type mechanical dryer, resulting to a product known as osmotically air-dried (OAD) semi-refined carrageenan. The blower (with specifications of ¼ hp, 220 volts, 50/60 cycles at 3000/3600-rpm, and Model SY-202 by Shin Yin Electric Works) discharged air at a rate of 0.06 m<sup>3</sup>/s. Weights of the osmotically air-dried (OAD) samples were determined every ten (10) minutes for the first one (1) hour and every fifteen (15) minutes thereafter. Drying was terminated when samples reached a moisture content of approximately 8-10%. Drying time, moisture reduction ratio, heat requirement, and power rating were computed subsequently. Three drying trials were done for samples osmotically dehydrated at 30<sup>o</sup>, 40<sup>o</sup>, and 50<sup>o</sup>C. Osmotically air-dried products were compared to purely mechanically air-dried (AD) samples.

ANOVA and Fisher's LSD Test were used to analyze results.

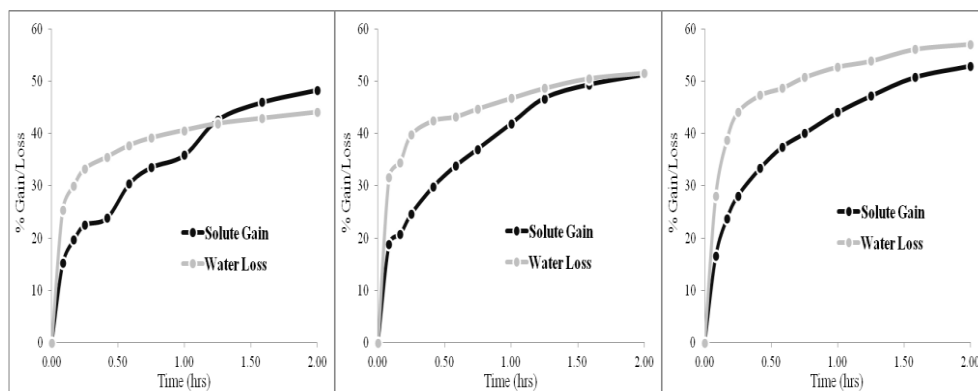
## **Results and Discussion**

### ***Mass Transfer Kinetics***

Dried raw seaweeds initially had a mean moisture content of 33.71%. Rehydration of the samples increased the moisture content to 83.55% and further to 94.62% upon cooking them in hot alkali solution. After subjecting to osmotic dehydration (OD) for two hours, moisture content was reduced to 33.54-47.82%. However, solids also increased dramatically from 6.41-6.84% to 52.18-66.46%.

As observed in the curves (Figure 1), there was high initial sucrose gain and water loss from the samples followed by slower rates in the latter stages. Rapid moisture removal and solute uptake in the beginning was apparently caused by the huge osmotic gradient between the hypertonic medium and the diluted moisture content of the modified seaweed. Over time, there was slight decrease in the osmotic pressure of the solution due to the simultaneous sucrose migration and incorporation of water from the samples, thereby slowing down both solute gain and moisture removal in the product. Furthermore, a protective subsurface deposit of sugar may also be formed in the food which acts as barrier against mass diffusion and impedes concentration gradients across the

product-medium boundary (Lazarides *et al.*, 1995). Structural changes due to collapse of surface cells and shrinkage may also take place, causing compaction of surface layers and enhanced resistance against mass transfer for solutes and water (Agarry *et al.*, 2008; Antonio *et al.*, 2008; Azoubel and Murr, 2004; El-Aouar *et al.*, 2006; Lazarides *et al.*, 1995; Silva *et al.*, 2012).



**Figure 1.** Solute gain and moisture loss during at different temperatures of 40, 50 and 60°C (left to right)

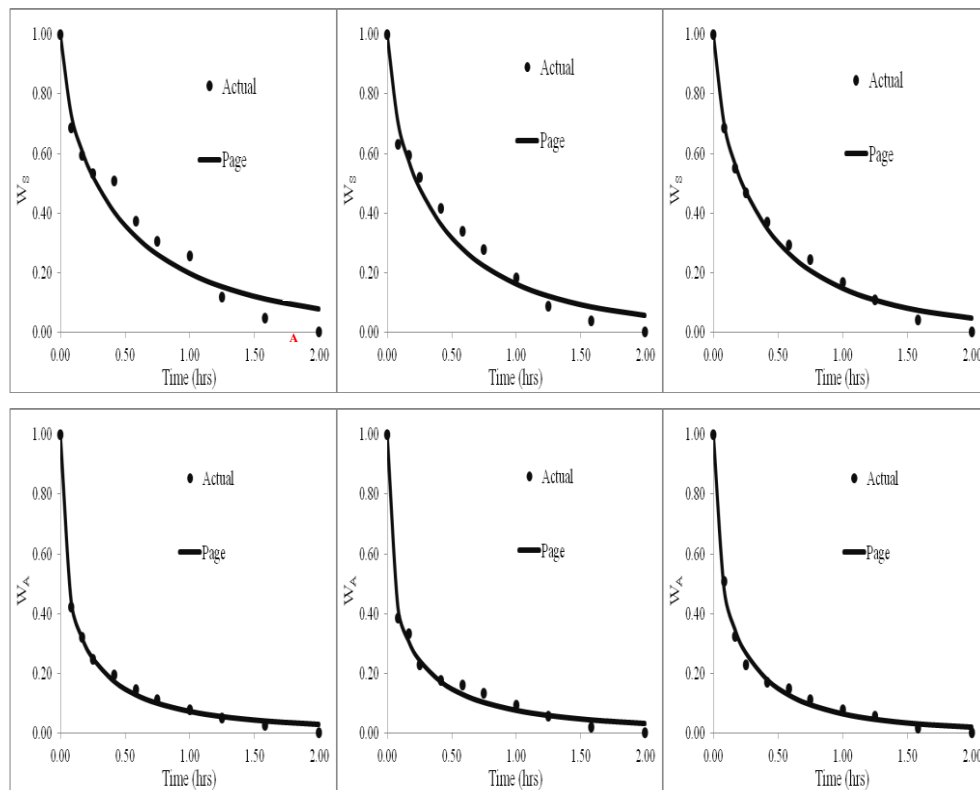
The plots in Figure 1 also show that water removal happened at a much faster rate than solute gain. This is because osmotic transport of water across semi-permeable cellular membrane is not as restricted as the passage of solids (Matuszek and Meresz, 2002).

### Mass Transfer Modelling

Drying model by Page was used to describe solute gain and water loss in the osmotically-dehydrated (OD) semi-refined carrageenan (Figure 2). Using the curves, factors  $k$  and  $n$  of the Page equation (Table 1) were determined; whereas, Table 2 summarizes the correlation results.

**Table 1.** Mean values of constants in the Page model

Temperature (°C)	Sucrose Gain		Water Loss	
	$k$	$n$	$k$	$n$
30	1.6319	0.6670	2.7850	0.4857
40	1.8271	0.6683	2.6434	0.4447
50	1.9141	0.6792	2.7665	0.5353



**Figure 2.** Page model of solute gain (top) and moisture loss (bottom) at different temperatures of 40, 50 and 60°C (left to right)

**Table 2.** Correlation between factors and constants in the model

Factor	Sucrose Gain		Water Loss	
	k	n	k	n
Temperature	0.566*	0.100	-0.138	0.091

Based on Table 2, only the constant  $k$  in the Page model showed positively strong correlation with temperature at 5% level of significance. Results of ANOVA also confirmed this observation. As to water loss, there was no clear association between temperature and the parameter, implying that the factor had no direct influence on moisture removal.

Generally, the results implied that temperature affected solute uptake but not water loss. In osmotic dehydration, the greatest driving force for mass mobility is the pressure gradient which in turns depends strongly on temperature and concentration of the osmotic solution (Rastogi, 1997). The

effect of the second factor was not significant since a high sucrose concentration solution was used in the experiment, as well as a high product-solution ratio was followed. Previous works (Herman-Lara *et al.*, 2013; Lazarides *et al.*, 1995) also showed increased mass transfer during osmosis at higher processing temperature. The increase in solid gain can be attributed to membrane swelling during osmosis and its plasticizing effect, which consequently improves cell membrane permeability to solute molecules (Ganjloo *et al.*, 2012). Furthermore, the osmotic solution also becomes less viscous at higher temperature, thereby enhancing mass diffusion on the food surface (Azoubel and Murr, 2003).

### *Air-Drying Parameters*

**Table 3.** Moisture and solid contents of products after osmotic dehydration and prior to air-drying

Treatment	Moisture Content (%) after OD	Solid Content (%) after OD	Solid Gain (%) Based on Page Model
T <sub>1</sub> (30°C OAD)	70.60	29.40	15.54
T <sub>2</sub> (40°C OAD)	67.21	32.79	18.06
T <sub>3</sub> (50°C OAD)	66.06	33.94	19.07

Processing time for osmotic dehydration was based on the allowable solid gain in the product. It is generally observed that addition of sugar to carrageenan generates stronger networks with a maximum strength at about 50% co-solute (Hill *et al.*, 1998). However, at high solids environment, the functionality of a polysaccharide effectively deteriorates (Hill *et al.*, 1998; Nishinari *et al.*, 1994). In other studies where sugar is mixed with carrageenan in low concentrations, the addition of sucrose at 12.5% (Nussinovitch, 2007) or 15% (Sabadini *et al.*, 2006) is proven to be sufficient enough to produce desirable results in terms of rheological properties of gel. To generate conservative results, 15% solid gain was considered in the final products. Using Page model, processing time for osmotic dehydration of the samples was estimated to be seven (7) minutes, time enough for the products in each treatment to attain at least 15% addition of sucrose (Table 3).

Drying properties (Table 4) of osmotically air-dried (OAD) samples at different osmotic temperatures were compared to mechanically air-dried (AD) samples.

**Table 4.** Air-drying data for AOD and AD products

Treatment	Drying Time (hr)	Moisture Reduction Rate (%/hr)	Heat Requirement (kJ)	Power Rating (kJ/s)
T <sub>1</sub> (30 <sup>o</sup> C OAD)	0.62 <sup>a</sup>	101.81 <sup>d</sup>	485.94 <sup>f</sup>	0.22 <sup>h</sup>
T <sub>2</sub> (40 <sup>o</sup> C OAD)	1.12 <sup>b</sup>	51.43 <sup>e</sup>	500.74 <sup>f</sup>	0.12 <sup>i</sup>
T <sub>3</sub> (50 <sup>o</sup> C OAD)	1.43 <sup>c</sup>	39.46 <sup>e</sup>	484.80 <sup>f</sup>	0.09 <sup>j</sup>
T <sub>4</sub> (AD)	1.67 <sup>c</sup>	50.87 <sup>e</sup>	857.26 <sup>g</sup>	0.14 <sup>i</sup>

Note: Same letters indicate that treatment means are not significantly different at 5% (LSD)

OAD samples at 30<sup>o</sup>C had the shortest drying time with a mean of 0.62 hour while untreated (AD) samples had the longest drying time of 1.67 hours. The disparity in drying time is expected as the treated samples had a lower initial moisture content than the control. However, as OD processing temperature increased, air-drying was also slowed down to a point that it was not statistically different from the control. This could be attributed to the slight difference in the sucrose gain of samples (Table 3). Higher processing temperature promoted greater uptake of sugar which consequently modified the composition of the final product. Aside from posing additional resistance to mass exchange, the subsurface layer of sugar created during the process also lowers the rates of complementary dehydration (Lenart and Grodecka, 1989; qtd in Azoubel and Murr, 2004; Rastogi *et al.*, 2014). The subsurface layer of sugar, especially when present in high amount, decreases vapor pressure of the surface (Rahman, 2012).

Similar behavior can be also observed in the moisture reduction ratio. Samples subjected to 30<sup>o</sup>C OD had the fastest rate at 101.81%/hr, which is significantly different from the other treatments. At 50<sup>o</sup>C, the moisture reduction rate (39.46%/hr) was observed to be much lower than the control (50.87%/hr).

In terms of heat requirement, measurements for OAD samples were statistically different to AD. This is due to the lower initial moisture contents of products subjected to osmotic dehydration which implies reduced amount of moisture to be removed and thus, lesser energy requirement. As to power rating, 30<sup>o</sup>C OAD proved to be more efficient with a mean value of 0.22 kJ/s which was significantly different from the other treatments. However, the process became less efficient at higher osmotic temperature. At 40<sup>o</sup>C OD, power rating was reduced to 0.12 kJ/s and to a much lower value of 0.09 kJ/s at 50<sup>o</sup>C. This is attributed to both the differences in the initial moisture content of the products and the drying time. At 40<sup>o</sup> and 50<sup>o</sup> OD, drying time is slowed



down because of higher sucrose gain than at 30°C. This greater concentration of sugar in the samples lessened the power rating despite the lower initial moisture content of the samples.

## Conclusions

Temperature is an important factor in the osmotic dehydration of semi-refined carrageenan. Higher temperature leads to greater uptake of solutes of the products, and vice versa. The deposition of sugar in the subsurface layers of the food material further influences its drying behavior in the subsequent air-drying. Osmotic dehydration could potentially reduce energy cost during air-drying since products' initial moisture content was reduced, thereby decreasing drying time. However, OD at higher processing temperature had less positive results due to higher sucrose gain which in effect lessened the drying rate and power rating

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