Effects of osmotic dehydration on the quality of semi-refined carrageenan

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***Abstract** – The study investigated the effects of osmotic dehydration (OD) as pre-treatment to the quality of mechanically air-dried semi-refined carrageenan (SRC). It aimed to evaluate some physical and textural properties of SRC as influenced by osmotic temperature. Dried seaweeds of the specie Kappaphycus alvarezii were used as raw material. After cooking and washing, the seaweed samples were subjected to osmotic dehydration in a saturated sugar solution at temperatures of 30O, 40O, and 50OC. The samples were then subjected to mechanical air-drying and milling to create powdered products. For property evaluation, both powder and gel samples were used. The osmotically air-dried products (OAD) were compared to conventionally-produced air-dried (AD) samples.*

Osmotic dehydration both induced moisture loss and solute uptake in the products. Due to sucrose gain, OD produced lighter powders and clearer gels from semi-refined carrageenan. Indeed, sugar was able to improve product clarity. OAD gels also had lower pH values, more viscous, and with greater strength than AD gels. In effect, this enhanced product qualities implied greater versatility and range of application for the semi-refined carrageenan, making it more competitive with its refined counterpart. However, osmotically air-dried (OAD) powders had higher water activity than purely air-dried (AD) samples. This is a challenge to the storability and shelf-life of the product, implying the need to develop appropriate packaging system for it.

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

INTRODUCTION

Seaweed is one of the major products of aquaculture. Widely recognized for its important ecological and economic values, it is being cultured mainly for consumption. In the Far East and Asian Pacific, seaweed is a traditional component of most diet while in western countries, it is utilized industrially as a source of hydrocolloids such as agar, alginate and carrageenan [1]. In the Philippines, seaweed is one of the most successful fisher products in recent years. The industry pegged export sales of US$615.9 million in 2013[2]. By 2016, it was projected to generate more domestic and export sales of $14 million and $394 million, respectively, by venturing to new markets in Asia, South America and Africa for quality seaweed-based products like raw dried seaweed, carrageenan and agar [3].

The utilization of seaweed as hydrocolloid is due to its polysaccharide contents which can be used as agent for enhancing gelling, viscosity, texture, and cell-mobilizing properties of various food, pharmaceutical, industrial and biotechnological products [4]. Semi-refined carrageenan (SRC), a type of hydrocolloid, is produced by heating seaweed in an alkaline solution of potassium hydroxide. In this method, the carrageenan is never exactly extracted from the parent material. Though production is easier and more practical, the SRC flour is generally colored, often has a high bacterial count and is not suitable for human consumption. This type of carrageenan contains cellulose that was in the original seaweed and gives cloudy solution which poses issues to product clarity [5]. Yet, product quality may be further enhanced to have acceptability at par with refined carrageenan. Osmotic dehydration may possibly address these issues.

Osmotic dehydration (OD) is a pre-treatment procedure commonly performed to food products prior to air-drying. The procedure involves submerging food in a hypertonic solution, and since the medium has higher osmotic pressure, there is partial migration of moisture from the food material, as well as solute ingress into the food tissues. Osmotic dehydration, which is successful even at ambient condition, can preserves the flavor, texture and color of food from heat, and is used as a pre-treatment procedure to enhance the sensorial, functional, and even nutritional properties of food [6].

The study examined the effects of osmotic dehydration on some properties of the semi-refined carrageenan. Due to the discoloration caused by the presence of cellulose and parent materials left behind after extraction, the final product has less economic value than its refined counterpart. In many cases, semi-refined carrageenan are not used for direct human consumption, limiting its use to non-food or non-human food products like pet food additives, and agents in cosmetic preparations. Sucrose, which was used as medium for osmosis, helps in improving the clarity of opaque gels [5]. The process employed in carrageenan extraction can also influence and produce variations in the primary structure of the product, affecting properties like thermal stability, elasticity, yield stress and optical clarity [7]. Thus, the improvements caused by osmotic dehydration on semi-refined carrageenan can possibly open the product for wider range of utility and application in the world market.

MATERIALS AND METHODS

2.1. Extraction of Semi-refined Carrageenan

Extraction of semi-refined carrageenan followed actual local industry practices in the Philippines. After acquiring dried seaweeds of the specie Kappaphycus alvarezii from local suppliers, they were soaked for 30 minutes in distilled water. The rehydrated seaweeds were then cooked in an alkali solution with the proportion of 22.4 g KCl and 300 g KOH dissolved in four (4) liters of water for every one (1) kilogram of seaweed. The sample were heated in the alkali solution at a temperature range of 80-85OC for approximately two (2) hours. After cooking, the seaweeds were thoroughly washed with distilled water to remove other components of the raw material. The remaining substance is referred to as the semi-refined carrageenan.

2.2. Osmotic Dehydration

After washing, the cooked seaweed was chopped into uniform length of approximately one (1) inch with average thickness of 2.50 mm. Seaweeds with weights of 5.10 ± 0.10 grams were then subjected to osmotic dehydration; in this procedure, samples were submerged in saturated sucrose solution at 30O, 40O, and 50OC. the osmotic temperatures served as the treatments for this research. Product-solution ratio was maintained at 1:20 since this is high enough to neglect concentration changes during the process [8]-[9]. The beakers containing the solution and samples were placed in a hot plate to ensure uniformity of solution temperature. The osmotic process lasted for seven (7) minutes.

After removing from the solution, the seaweeds were washed quickly in a pan of distilled water and then gently blotted with adsorbent paper to remove excess solution and sugar adhering to the surface. Pressing and squeezing of samples was avoided. Weights were then measured. All experiments were done in triplicates. Moisture content of the samples was then determined using oven (CENCO air oven) method.

2.3. Air-Drying of Samples

To produce the SRC powder, osmotically-dehydrated samples was subjected to mechanical air-drying at 60OC using a laboratory-type mechanical dryer with a blower discharging air at 0.06 m3/s. The oven was initially run for fifteen (15) minutes or until temperature inside the chamber stabilized. 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) was set to full open in all runs. Drying was terminated when samples reached a moisture content of approximately 8-10% which was monitored using the moisture balance principles.

Dried samples from the semi-refined carrageenan subjected to different osmotic temperatures, also referred here as osmotically air-dried (OAD) samples, were compared to dried samples which had not undergone osmosis, referred here as purely air-dried (AD) samples.

2.4. Product Evaluation

After air-drying, samples were pulverized into fine powders using kitchen-type food mill. Powdered samples were then tested for color and water activity. Color of the SRC powder was determined based on lightness or L* value in the CIEL*a*b* using Konica Minolta Colorimeter BC-10. The test material was placed inside a clear plastic container before the instrument's conical eyepiece was lightly positioned on it and readings were taken. Ten (10) measurements were made for each treatment. On the other hand, water activity was evaluated by acquiring the diagnostic services of DOST Region IV-A CALABARZON. Fifty (50) grams of each treatment was submitted for measuring water activity using Novasina water activity meter. Three (3) readings were done for each treatment. Gel products at 1.5% carrageenan solution were made from the powder. Color of 5-mm thick gels (laid on white background) was evaluated using the Konica Minolta Colorimeter, employing the L* value of CIEL*a*b* scale. Ten (10) measurements were made for each treatment. Values of pH at 30OC were measured using a pen-type digital pH meter. Six (6) pH readings on different samples per treatment were made. All pH measurements were first converted to [H+] before calculating the mean [10] in order to have valid symmetrical confidence limits. These values were then reconverted to pH units for further statistical tests. Conversion of pH to [H+] is given as pH = - log[H+]. Furthermore, viscosity was evaluated at 75OC using Viscometer Rion with its spindle 2 rotating at approximately 62.5 rpm. Viscosity was measured in terms of poise and three (3) replications were done per treatment. For measuring strength, gels were stored overnight at 5OC to further stabilize the gels. After which, samples were subjected to puncture test. The test was performed using a 5-kg load cell equipped with an 8-mm probe of Instron 4400 with crosshead speed of 1 mm/s at a depth of 15 mm. Five (5) samples were prepared per treatment and the probe was penetrated into each sample four times, allowing a relaxation period of at least 30 sec between penetration cycles.

2.5 Statistical Analysis

The study followed Completely Randomized Design (CRD) and results were examined using Analysis of Variance (ANOVA). To satisfy the assumptions of ANOVA, Shapiro-Wilk test was performed to determine if data were normally distributed and Levene's test of homogeneity to determine if variances were more or less equal. Since data came from small sample size, minor violation of the assumptions was acceptable. If ever data failed either of the two tests, transformations were made. Microsoft Excel and SPSS 17.0 were used to perform ANOVA and the subsequent post-hoc tests (using Least Significant Difference) on the test results for the OAD and AD semi-refined carrageenan.

RESULTS AND DISCUSSION

The moisture and solid contents after seven (7) minutes processing time for the osmotically air-dried (OAD) semi-refined carrageenan are given in Table 1. The solute gains of the samples were estimated using Page model. In several studies where sugar is incorporated into carrageenan at low concentration, the addition of 12.5% sucrose [11] or 15% [12] is found to produce desirable physical and rheological properties of gel. However, the functionality of a polysaccharide deteriorates at high solids environment [13]-[14].

Table 1. Moisture and solid contents of products after osmotic dehydration and prior to air-drying

Treatment	Moisture Content (%) after OD	Solid Content (%) after OD	Solute Gain (%) after OD
T_1 (30^{O}C OAD)	70.60	29.40	15.54
T_2 (40^{O}C OAD)	67.21	32.79	18.06
T_3 (50^{O}C OAD)	66.06	33.94	19.07

Some physical and textural qualities of OAD samples were compared to purely air-dried (AD) products. Results of property testing are summarized in Table 2.

As observed, color values of osmotically air-dried (OAD) semi-refined carrageenan powders were significantly different from the control (AD). The process indeed prevented darkening of samples during air-drying, thereby producing products lighter in color. Similar results were observed in other food materials. Osmotic dehydration prevented color damages in apple, banana, potato and carrot [15] so that treated samples did not brown as much as the untreated samples and the lightness decreased only slightly. Sucrose is a non-reducing sugar itself and would not undergo non-enzymatic browning [16]. Loss of color during osmotic dehydration could also be due to degradation or loss of concentration of food pigments [17].

Gel color of treated samples was statistically different from AD samples. Hence, osmotic dehydration was able to produce gels with more clarity, possibly because of the infusion of sugar in the product. In other food materials like gellan gels, adding sucrose also increased gel clarity [18]. Sucrose can indeed increase gel clarity by reducing the differences in refractive index between polymer and the medium [18]-[19].

Table 2. Properties of semi-refined carrageenan powder and gel products

Treatment	Powder		Gel			
	Color (L*)	Water Activity	Color (L*)	Viscosity at $75^{O}C$ (cps)	pH at $30^{O}C$	Gel Strength (kN/mm^2)
T_1 ($30^{O}C$ OAD)	78.58^{a}	0.736^{d}	56.90^{h}	76.67^{j}	8.01^{m}	4.70^{o}
T_2 ($40^{O}C$ OAD)	80.98^{b}	0.712^{e}	57.28^{h}	146.33^{k}	7.97^{m}	6.98^{p}
T_3 ($50^{O}C$ OAD)	81.51^{b}	0.633^{f}	59.23^{h}	216.67_{l}	8.02^{m}	9.14^{q}
T_4 (AD)	57.61^{c}	0.398^{g}	52.25^{i}	56.33^{j}	9.19^{n}	1.65^{r}

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

In simpler terms, sucrose lessens the optical contrast between gel and the surrounding environment. Sugar by itself cannot increase the clarity of water solution; in fact, the sugar solution is slightly more turbid than pure water, possibly due to light scattering effect of sucrose molecules.

When compared with polymer gels, even the light scattering from the solution is negligible. Therefore, although sugar molecules may scatter light, their contribution to the turbidity in a gel system is too small compared to the turbidity resulting from gelation of gel components. In effect, the turbidity of the entire system is reduced, as well as the optical contrast between the polymer and the medium.

Moreover, the higher viscosity of sugar solutions might also contribute to gel clarity because viscose media hindered the growth of junction zones in the gel [18]. During gelation, double helices migrate and become orderly packed into existing junction zones. Increasing the viscosity of the medium hinders such migration and causes the size of the junction zones of sugar gels to be smaller than that of water gels. Therefore, sugar gels are clearer than corresponding water gels.

Statistically, water activities of products were significantly different for each treatment. The results indicate that at 8-10% moisture content, OAD products have higher water activity than the control. This can be expected as osmotic dehydration essentially produces food products that belong to the class of intermediate moisture food with water activity ranging from 0.65 to 0.90 [20]. This could be attributed to the sucrose content of the osmotically-dried products. Sugar is a hygroscopic material and has high water-binding capacity, referred to as humectancy [21], which consequently increases water activity of food materials.

Among the treated products, there is decreasing water activity with increasing osmotic temperature. It is a result of the greater sucrose gain in higher temperature processing. Generally, water activity is decreased with increasing sucrose concentration [16],[22].

It has been reported that osmotically air-dried foods are unstable during storage [20] because of their relatively high water activity. Yet, the a_w values for the osmotically-dried carrageenan are still within safe limits. Water activity of 0.93 is enough to suppress the growth of most pathogenic bacteria (with the exception of *Staphylococcus aureus* which grow aerobically at a_w values down to 0.86), while most mold and yeast strains are inhibited between 0.88 and 0.80 [23].

Moreover, at higher osmotic temperature, there was a significant increase in the viscosity of solution. It is possibly because of the infusion of solute in the carrageenan since sucrose generally increases viscosity values [22]. Furthermore, osmotically dried samples exhibited a viscous rather than an elastic behavior, indicating that the infusion of sugars causes plasticity in the food structure [24].

The mean pH values for the OAD samples were significantly different from that for AD. Thus, osmotic dehydration significantly reduced the pH values of the final products, making them less alkaline. After extraction where raw material was cooked in hot alkali solution, the carrageenan product was expected to have high pH values as KOH is a strong base. Since sucrose is introduced into the product during osmotic dehydration, concentration of alkaline components became diffused, lowering the pH values.

Lastly, the gel strength for the OAD samples was significantly different from AD gels. Thus, osmotic dehydration significantly strengthened carrageenan gels, with $50^{O}C$ processing temperature yielding the strongest gels. This could be attributed to the sucrose gain of the OAD products. Sugar is found to increase firmness in gels because of its good water-holding capacity [25]. Sucrose also exhibits a stabilizing effect on formation and packing of carrageenan double helices [18]-[19]. Therefore, increased sucrose concentration via osmotic dehydration potentially increases strength of carrageenan gels

CONCLUSION AND RECOMMENDATION

Osmotic dehydration of semi-refined carrageenan resulted to powdered product with lighter color and gel with better quality, reasonably because of sucrose gain. Other desirable properties of osmotically air-dried (OAD gels relative to purely air-dried (AD) samples were lower pH values, greater viscosity, and greater strength. Such properties were generally enhanced at higher osmotic temperature which resulted to higher sucrose uptake.

The results showed that osmotic dehydration indeed improved the quality of semi-refined carrageenan, both as a powdered product and a gel. In powdered form, it was lighter in color, making it more attractive and desirable in more applications, attaining more competitive stand with the refined product. In gel form, its enhanced viscosity and strength implies better emulsifying and texture-modifying properties, especially in human food preparations like in meat and dairy. In the Philippines where more local producers are engaged in semi-refined production because it is comparatively easier, the results would add more physical and economic value to their final product.

However, osmotically air-dried (OAD) powders had higher water activity than purely air-dried (AD) samples which may challenge product storability. Thus, moisture sorption of OAD semi-refined carrageenan can also be studied, as well as appropriate packaging technologies. Lastly, one of the major by-products of carrageenan extraction is the broth drained from the cooked seaweeds. The solution is rich with soluble protein, carbohydrates and other components removed from the seaweed. It is recommended to aid in further research for the utilization of such by-product.

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