A novel ohmic-based technology for seaweed processing

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Abstract

Carrageenans are important hydrocolloids extracted from red seaweeds (Rhodophyceae) of genus *Eucheuma, Iridiae, Gigartina,* and *Chondrus*. Carrageenan extraction technologies currently applied in modern industries are based on conventional heating using double-jacketed tanks supplied with steam as heating medium. This technology is inefficient in energy use and posesa complicated processing setup since it requires a dedicated steam generator and steam delivery system. The objective of this study was to develop an ohmic-based novel technology for seaweed processing. The system developed and used in this study consisted of three ohmic heating chambers (7.5 liter each) working in parallel in static mode. The system was tested for iota carrageenan extraction from *Eucheuma denticulatum* (known commercially as *Eucheuma spinosum*) using 0.3M KOH solution as extracting medium with seaweed to solution ratio of 1:35 (w/v).The tests were conducted at several temperatures and extraction durations to gain broad insights on the effectiveness of this novel technology. The test results indicated that this technology can efficiently extract carrageenan from *E. spinosum* with yields in the range of 41-61% (dry weight basis) and viscosity and gel strength in the range of 269-274 cP and 50.87-55.27 g/cm^2 respectively. These results suggest that ohmic-based seaweed processing technology is a viable alternative to conventional technologies currently applied in industries.

Introduction

Carrageenans, agar-agar, and alginate are phycocolloid found in several classes of seaweeds. Carrageenans and agar-agar are commonly found in red-algaeof the classes Rhodophyceae andFlorideophyceae while alginate is found in brown algae of the class Phaeophyceae. Carrageenans are produced from *Kappaphycus* spp (Santos, 1989; de Ruiter and Rudolph, 1997; Webber *et al*., 2012; Orbita, 2013; Hernandez-Carmona *et al*., 2013), *Eucheuma* spp (Santos, 1989; de Ruiter and Rudolph, 1997; Viana *et al*., 2004;Freile-Pelegrin *et al*., 2006; Hernandez-Carmona *et al*., 2013; Villanueva and Montano, 2014), *Hypnea* spp (Ramalingam *et al*., 2003; Mtolera and Buriyo, 2004; Diaz *et al*., 2010; Al-Alawi, 2011; Arman and Qader, 2012; de Oliveira Farias, 2014) and *Chondrus* spp. (Mathieson and Tveter, 1975; de Ruiter and Rudolph, 1997; Bixler and Porse, 2011; Hernandez-Carmona *et al*., 2013). Agar-agar is produced from redseaweeds of genus *Gelidium, Gelidiella, Pterocladia, and Gracilaria*(Doty and Santos, 1975; Hurtado-Ponce, 1992a,1992b; Bixler and Porse, 2011; Hernandez-

Carmona *et al*., 2013; Croce *et al*., 2015) and alginates are mostly produced from *Laminaria* spp., *Macrocystis pyrifera, Ascophyllum Nodosum, Sargassum, Ecklonia,* and *Lessonia* (Bixler and Porse, 2011; Hernandez-Carmona *et al*., 2013).Many of these commercially important seaweeds have been successfully cultivated.

Global production of cultivated seaweeds exceeded 23 million tons in 2012 with an estimated value of approximately 6.37 billion USD (FAO, 2014). Statistical data reported by FAO (2014) also indicated that the main producers of cultivated seaweeds were Asian countries (99.18%) led by China (12.83 million mt), Indonesia (6.51 million mt), and the Philippines (1.75 million mt). Indonesia and other countries in the coral triangle region produced mostly *Kappaphycus, Eucheuma,* and *gracilaria* while other typeswere produced in smaller quantities (Hurtado *et al*., 2014).

Carrageenan producing seaweeds (carrageenophytes) can be processed into semi-refined carrageenan (SRC) via alkalization and refined carrageenan (RC) through extraction. These processesare generally done in alkali solutions such

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as KOH and NaOH. Heating of seaweeds in alkali solution causes imbibitions of thealkali solution into the seaweed which can modify the properties of carrageenans contained in the processed seaweed through desulfation.

Inthe mid 1970s, processing of carrageenan containing seaweeds without extracting the carrageenan from the seaweed was started in the Philippines (Bixler and Porse, 2010). Since then, this processing method has been gaining popularity due to the fact that it is much lower in production costs since energy consumption is lower and no precipitation is required in the production process. This processing methodis done by heatingthe seaweeds in relatively higher alkali concentration (8 – 12% KOH solution) and at temperature below 80oC (below melting temperature of the carrageenan) such that the carrageenansremain in the seaweed. The use of lower temperature in this process is intended to lower carrageenan loss which can occur due to leaching of the carrageenans into the heating solution. Following the alkalization process, the seaweedsare coarsely chopped, dried, and grinded. This process is generally used for processing of Kappaphycus alvarezii and the product produced from this process is referred to as semi refined carrageenan (SRC), processed eucheuma seaweed (PES), Philippines Natural Grade (PNG), alkali treated cottonii (ATC), or alkali modified flour (AMF).

Prior to the introduction of the semi refined process indicated previously, carrageenan production through extraction was the only processing method employed in seaweed processing industries. This processing method is done by heating seaweeds containing carrageenan at temperatures above 90oC (above the melting temperature of the carrageenans).Heating at thishigh temperature can effectively disintegrate the seaweed structure and extract the carrageenan out of the cell wall and cell matrices. To recover the carrageenan from the solution, subsequent processes which include filtration, precipitation in alcohol or KCl solution (for kappa carrageenan), drying, and grinding are generally done. The carrageenan obtained from this whole process is commonly known as refined carrageenan (RC).

The chemical process involved in the modification of carrageenan properties and extraction of carrageenan from seaweeds is relatively simple (McHugh, 2002). However, as McHugh (2002) pointed out,technological and engineering aspects of the processing technologyare quite complex, especially for RC production.

Many studies reported in literatureshave attempted to elucidate the factors that affect yield and quality of carrageenans produced from the alkalization and extraction processes. Extraction of kappa carrageenan and kappa-iota hybrid has been studied by Ohno *et al*. (1994, 1996), Hoffmann *et al.* (1995), Rideout and Bernabe (1998), Normah and Nazarifah (2003), Munoz *et al.* (2004), Tuviken*e et al.* (2006), Montolalu *et al.* (2008), Bono *et al*. (2014) and Webber *et al*. (2012). Studies on extraction of iota carrageenan from different types of seaweeds have also been reported (Istini *et al*., 1994; Viana *et al*., 2004; Villanueva *et al*., 2004; Araujo *et al*., 2012). In all of the aforementioned studies,the extraction process were conducted using conventional heating method, i.e. heating of seaweeds and extracting medium on a stove or a hot plate.

Productions of carrageenan, agar, and alginate in seaweed processing industries are generally done by heating the processed seaweeds in double jacketed tanks supplied with steam as a heating medium. This method of heating requires a dedicated steam generator and steam delivery system such that the processing system can become quite complex. In this study, aninnovative technology based on ohmic heating phenomenon was developed for seaweed processing. This technology can be used for the semi refined process (alkalization process) and for extraction of carrageenan, agar-agar, and alginate from various types of seaweeds.

Ohmic heating is an internal heat generation due to the resistance of a material to electric current flow. The concept of heating due to the passage of electric current through an electrically conducting material has been known since James Prescott Joule (1818-1889) introduced it in 1840 (Sastry *et al*., 2002). Therefore, the phenomenon of internal heat generation in a material due to dissipation of electrical energy into heat is known as Joule heating or ohmic heating.

In the area of food processing, ohmic heating is generally defined as a process whereby heat is generated internally within food materials (liquids, solids, or a mixture of both) due to the passage of electric current through the food materials. This internal heat generation is caused by the electrical resistance of the food materials (Sastry and Barach, 2000). The amount of heat generated internally within the food material is directly related to the electrical conductivity of the processed food and the density of electric current generated by the applied electric field.

The internal heat generation during ohmic heating can be used to heat food materials up to the desired temperatures (Salengke and Sastry, 2005; 2007a, 2007b). In addition, ohmic heating has been found to

provide rapid and uniform heating of food materials provided that they have uniform and moderate to high electrical conductivities (Salengke and Sastry, 2007b;2007c). Therefore, ohmic heating technology can be used effectively in food processing operations requiring rapid and uniform heating.

Ohmic heating process can be very efficient in energy use since all of the electric energy consumed in the process can be converted into heat.This heating method has been used in many food processing operations and one potential application that needs to be explored is the use of ohmic technology in seaweed processing. This heating method can be applied in alkalization of seaweed to produce semi-refined carrageenan and in extraction of important food hydrocolloids such as carrageenan, agar, and alginate from seaweeds.This is due to the fact that these processes are typically done in KOH or NaOH solutions which have high electrical conductivities. Under such condition, rapid and uniform heating can be achieved via ohmic heating process.Therefore, the objectives of this study were to develop a new and innovative technology for seaweed processing based on ohmic heating and to examine the performance of this technology for heating of KOH solution and extraction of carrageenan from Eucheuma spinosum.

Materials and Methods

The ohmic heating system developed in this study consisted of three main components, i.e. ohmic heating chamber, electric supply and control system, and data acquisition system. The ohmic heating chamber was made of a PVC pipe (internal diameter 10.25 cm and length of 100 cm). Stainless steel electrodes were secured at both ends of the PVC pipeto supply electric current to the ohmic heating chamber.The supply of electric current to the electrodes was controlled by a thermocontroller. Current, voltage, and temperature data were measured every second and recorded using a data logger (Campbell Scientific CR1000, Logan, Utah, USA). The current and voltage data were used to calculate the electrical conductivity of the solution within the ohmic heating chamber and power consumption during heating.

Testing of the ohmic heating system

The technology developed was first tested for heating of alkali solutions without seaweeds to assess the heating rate, electrical conductivitiesof the solutions, and electrical energy consumed during heating.The electrical conductivities of KOH solutions(0.3, 0.6, and 0.9 M) during ohmic heating were determined using Equation 1.

$$\sigma = \frac{I}{V} \cdot \frac{L}{A} \qquad (1)$$

In the above equation, σ represents the electrical conductivity (S/m) of KOH solutions during ohmic heating, I is the amount of current (Amps) which passed through the ohmic heating chamber, V and L are voltage difference (Volt) and distance (m) between the two electrodes, and A is the cross section area (m^2) of the ohmic heating chamber.

Another important aspect that needs to be measured during ohmic heating is power consumption. Electric power consumption during ohmic heating (P) can be calculated using Equation 2.In this equation, t represents time interval between measurements in second. The efficiency of electrical energy usage during ohmic heating is high since all of the electric energy is converted into heat. In this study, the efficiency of electrical energy consumption was estimated based on the amount of electrical energy consumed during heating (Equation 2) and the theoretical energy required to heat KOH solution, Q, (Equation 3). The energy efficiency was then calculated using Equation 4.

$$P = I \cdot V \cdot t \qquad (2)$$

$$Q = m \cdot cp \cdot \Delta T \qquad (3)$$

$$\eta = \frac{Q}{P} \times 100\% \qquad (4)$$

Carrageenan extraction

The technology developed was tested for extraction of carrageenans from Eucheuma spinosum(contains mostly iota carrageenan). Dry seaweeds (moisture content 30 – 35%) were obtained from seaweed farmers in South Sulawesi, Indonesia. The seaweeds were first cleaned from sands and salts and then rinsed with tap water to thoroughly clean the seaweeds. Thereafter, the seaweeds were sun dried to about 10% moisture content. Seaweed samples for extraction were prepared by weighing the dry seaweeds to predetermined weights and placed in sealable plastic bags and stored at room temperature. The samples were soaked in tap water for 30 minutes before extraction.

Extractions were conducted at three temperatures (85, 90, and 95°C)and three durations (120, 180, and 240 minutes). Extractions were conducted using 7.5 litre ohmic heating chambers. KOH concentration was maintained at 0.3M and the ratio between the seaweed and KOH solution was1:35 (w/v). After extraction process, the solution was filtered using cheese clothes to separate solid residues from the

extracting medium.

The extracted carrageenan contained in the solution was recovered through a precipitation process using 90% isopropyl alcohol. The carrageenan containing solution was added slowly into the alcohol while stirring to avoid formation of large lumps of the precipitated carrageenan. The final ratio between the alcohol and the solution was 2:1. The mixing was continued for 30 minutes after all of the solution was added into the alcohol to allow the carrageenan in the solution to precipitate completely. After that, the carrageenan was recovered by filtration using cheese cloths and cut into small pieces before drying. The drying was done via sun drying to moisture content of about 12%.

Carrageenan yields were calculated asthe ratio between the weights of the carrageenan produced (at 12% moisture content) and the dry weight of the seaweed processed. The quality of the carrageenan was determined based on solution viscosity and gel strength. Carrageenan solutions were prepared by dissolving 6 g of carrageenan into a glass beaker containing 400 ml water (1.5% carrageenan solution). The opening of the beaker was sealed using aluminum foil and the solution was heated on a hot plate while stirring using a magnetic stick until all of the carrageenan dissolved. The glass beaker was then inserted into a water bath maintained at 75°C. After the solution temperature stabilized at 75°C, the beaker was transferred to a viscometer (Brookfield Engineering, Middleboro, Massachusetts, US) for viscosity measurement. After the viscosity measurement, the carrageenan solution was recovered and cooled to room temperature in 200 ml beakers to form gel. The temperature of the gel was allowed to stabilize at room temperature for at least two hours before gel strength measurement was conducted. Gel strength of the carrageenan samples (expressed as Bloom value) was measured using a texture analyzer (Stable Micro Systems, Surrey, UK) equipped with Bloom test apparatus.

Results and Discussion

Heating rate, electrical conductivity, and energy consumption

Alkali solutions such as KOH and NaOH are commonly used in seaweed processing to increase the gel strength of carrageenans contained in the processed seaweed. These solutions have relatively high electrical conductivity at low concentrations (<1M) such that their ohmic heating rates are high. Data shown in Figure 1 reveal that ohmic heating rates of dilute KOH solutions (concentration 0.3 –
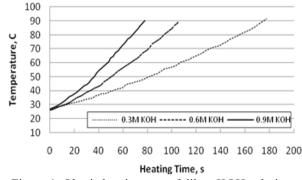

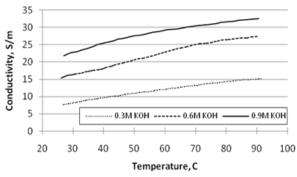Figure 1. Ohmic heating rate of dilute KOH solution.

Figure 2. Electrical conductivity of dilute KOH solutions.

0.9M) were relatively high. The typical processing temperatures used in carrageenan, agar, and alginate production can be reached within three minutes of heating using low concentration of alkali.At higher alkali concentrations such as those typically used in SRC production, the time of heating required to reach processing temperature will be much less. It is also important to note that voltage gradient inside the heating chamber was low;around 4.13 V/cm at the start of heating and down to 3.65 V/cm at the end of heating. This was due to the significant increase in current flow through the ohmic heating chamber as temperature, and thus electrical conductivity, of solution inside the ohmic heating chamber increased. The fast heating obtained from this study clearly indicates that ohmic based technology can be a viable technology for seaweed processing.

Electrical conductivity of dilute KOH solutionis highly dependent on concentration and temperature. The electrical conductivities of KOH solutions at concentrations of 0.3, 0.6, and 0.9M were about 8.15, 16.36, and 22.78 S/m respectively at 30°C, and 15.09, 27.30, and 32.61 S/m respectively at 90°C (Figure 2).These results are in accordance with the trend reported by Kortum (1965) which indicated that the electrical conductivities of electrolytes increase with the increase in temperature. Results of studies reported by Guanti and Moran (1986),See and White (1997),and Gilliam *et al.*(2007) showed that electrical conductivities of KOH solutions at high

concentrations increase linearly with temperature. Therefore,it is expected that the electrical conductivity of dilute (<1M) KOH solution used in this study during ohmic heating is linearly dependent on temperature as well.

The results of measurements shown in Figure 2 indicate that the curves for the relationship between electrical conductivity and temperature are slightly concave but the electrical conductivity values can still be approximated adequately using linear equations with R^2 values greater than 0.96. The slightly concave electrical conductivity curves observed in this study might indicate distortions to current flow due to bubbles and gas formations(most likely from the electrode surface). Nonetheless, theelectrical conductivity values found from this study were close to the calculated values reported by Gilliam *et al.* (2007). It is important to note that unlike the methods used by several researchers (Guanti and Moran, 1986; See and White, 1997; Gilliam *et al.*, 2007), the electrical conductivities of KOH solutionsfound in this study were determined by continuously measuring the amount of current flows and voltage different between the two electrodes inside the heating chamber during ohmic heating.

Total cumulative energy consumptions during ohmic heating of 4.35 kg of KOH solutions from 26 to 90oC as computed using Equation 2 were 390.19, 385.67, and 352.07 Watt-hour for 0.3 M, 0.6 M, and 0.9 M KOH solution respectively. The difference in the cumulative energy consumption was mainly due to the difference in the duration of heating time required to reach the final temperature of 90°C as shown in Figure 1. The amount of heat loss from the ohmic heating chamber to the surrounding is expected to be higher as the heating duration increases. The efficiencies of electric energy usage for ohmic heating of KOH solutions were 82.96, 83.93, and 91.94% for 0.3 M, 0.6 M, and 0.9 M solution respectively. It is important to note that the ohmic heating chamber used during measurements was not thermally insulated such that the energy loss to the surrounding was considerable. Therefore, in subsequent extraction experiments, the heating chamber was thermally insulated with glass wool to reduce heat loss during extraction.

Carrageenan yields

Carrageenan yields from various extraction conditions as shown in Figure 3indicate that average yields varied from 46 to 69% (at 12% moisture content) or equivalent to 41-61% (dry weight basis). These values are in accordance with average yield of 57.9% reported by Dawes *et al.* (1977), 44-
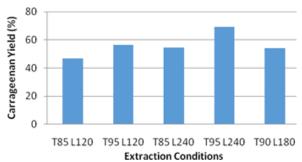

Figure 3. Carragenan yields from extraction of carrageenan from *E. spinosum.* T=extraction temperature (C), L = extraction duration (minute).

59% reported by Buriyo *et al.* (2001), 53 and 56% reported by Vairappan *et al.* (2003), 56.6% reported by Wakibia *et al.* (2006), and 43.5 – 57.2% reported by Freile-Pelegrin and Robledo (2008). These results clearly indicate that the use of ohmic heating technology in extraction of carrageenan, agar, and alginate from seaweeds is promising. This due to the fact that besides the desired rapid heating as shown previously; ohmic heating can also provide additional effectssuch as cell wall electrophoresis and tissue disintegrations as alluded previously by Lima and Sastry (1999), Wang and Sastry (2002), and Salengke and Sastry (2005).

The results obtained from this study clearly indicate that ohmic heating technology has great potentials in seaweed processing. This novel seaweed processing technology can provide not only higher extraction yield but also higher energy efficiency since heat is generated internally by the processed materials. The quality of carrageenan produce from this extraction method is good based on the viscosity value. The viscosity of 1.5 % carrageenan solution at 75oC ranged from 269.15 to 274.34 cP (Figure 4). These values are higher than the values reported from the study by Vairappan *et al.* (2003) which indicated average viscosity of 250 – 254 cP and the maximum viscosity of 161.3 cP and 144.6 cP reported by Freile-Pelegrin *et al.* (2006) and Freile-Pelegrin and Robledo (2008) respectively for iota carrageenan extracted from *Eucheuma isiforme.*The gel strengths of the carrageenan extracted from *Eucheuma spinosum* at 1.5% carrageenan concentration ranged from 64.41 – 69.98 Bloom value (Figure 5) or equivalent to 50.87 – 55.27 g/cm². These values are slightly higher than the values (< 50 g/cm²) reported by Freile-Pelegrin *et al.* (2006, 2007). However, the gel strengths found in this studywere considerably lower than the values reported by Vairappan *et al.* (2003) for carrageenan extracted from *Eucheuma spinosum.* The data reported by these authors indicated that the gel strengths varied greatly during the year where maximum gel strength
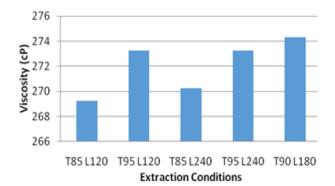
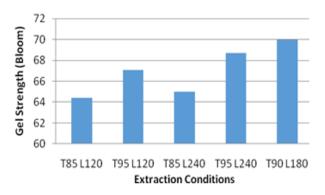Figure 4. Viscosity of carragenan extracted from *E. spinosum*

Figure 5. Gel strength of carragenan extracted from *E. spinosum*.

of 290 g/cm^2 was achieved in November and about 120 – 140 g/cm^2 in other months. It is important to note also that the gel strengths reported by Vairappan *et al.* (2003) was for gels containing 3% carrageenan.

The overall results of this study indicate that ohmic-based seaweed processing technology is a viable alternative to conventional technologies currently applied in seaweed processing industries. Extraction yields and viscosity of the carrageenan produced are high. However, further studies using pilot or industrial scale prototypes and other types of seaweeds are necessary to be able to fully evaluate this novel technology. In addition, processing parameters need to be optimized to improveyields and properties (viscosity and gel strength) of the carrageenan produced.

Conclusion

An ohmic-based technology for seaweed processing has been successfully developed and tested for extraction of carrageenan from *Eucheuma spinosum*. The overall results indicate that ohmic-based seaweed processing technology is a viable alternative to conventional technologies currently applied in seaweed processing industries. Extraction yields and viscosity of the carrageenan produced are

high but further testing and optimization of processing parameters need to be done to be able to fully evaluate the performance of this new technology.

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