Change of solvent proportion effect on pilot plan scale extraction of alginate from *Sargassum* sp.

R Kusumawati¹, J Basmal¹ and B S B Utomo¹

¹Research Centre for Marine and Fisheries Product Processing and Biotechnology, KS Tubun – Petamburan VI, Jakarta, Indonesia

Corresponding author’s e-mail : tanjung.1979@gmail.com

Abstract. Alginate extraction from *Sargassum* sp. pilot plant scale has been done. The extraction technique used refers to the modification of laboratory scale extraction techniques using acidic methods. On a scale of 1 kg of raw material, modification of the extraction technique was carried out on the proportion of bleaching material, sodium hypochlorite (NaOCl), which was originally 4% (v/v) to 2% (v/v). On a scale of 3 kg of raw material, modifications are made to the volume of water for extraction, which was originally 30 times the weight of the raw material to 20 times the weight of the raw material. On a scale of 10 kg of raw material, modifications are made to the ratio of water binder, iso propyl alcohol (IPA), which was originally 8 times the weight of the raw material to 3 times the weight of the raw material. The results of the study show that modification of the solvent carried out at each production scale causes an increase in yield (17.97, 19.79, and 22.67%) and a decrease in viscosity (128.43, 55.03, and 40.27 cPs).

1. Introduction
Indonesia consumes about 1,650 tons of alginate every year which was entirely supplied from imports[1]. The imported alginate was used in textile industry (50%), food (30%), paper production (6%), welding rods production (5%), and the other for pharmaceutical purposes (5%)[2].

There had been several research reports on alginate extraction in upscale level using Kelp *Macrocystis pyrifera* as raw material and acid method followed by additional of formalin solution [3-7]. Research for alginate production at laboratory level in Indonesia commonly used *Sargassum* sp. as a raw material related with the resource of *Sargassum* sp. is still abundance in Indonesia. The experiments in pilot plan level on alginate extraction using *Sargassum* sp. as raw material is still limited in Indonesia. Extraction using acid added with calcium chloride was chosen in in this study to saving production time. Characteristics of alginate such as viscosity is a specific character of alginate that can indicate the functional properties of alginate in commercial products. It is opportunities for research, especially using *Sargassum* sp. harvested at Binuangeun Beach, Banten, as raw material for alginate extraction.

Indonesia is a second country with the longest coast line in the world, having a high brown seaweed biodiversity, such as *Sargassum* sp and *Turbinaria* sp. Brown seaweed, as a raw material for alginate extraction, can be found in some Indonesian coast lines. One type of brown seaweeds which grow predominantly in Indonesia is *Sargassum* sp. That is not similar to *Eucheuma cottonii* which has been cultured successfully cultured in many areas in Indonesia[8], *Sargassum* sp. has not been cultured...
intensively. Yunizal et al (2000) has successfully cultured *Sargassum* sp. in Binuangeun Beach, Banten [9]. The successful small scale culture experiment should be developed and implemented immediately to support sustainability of raw material for alginate industry development. Method of alginate extraction from brown seaweed need to be conducted in order to develop alginate industry Indonesia, improving alginate extraction method resulting in good quality product and in return it is able to support raw material sustainability.

2. Materials and methods

This study used *Sargassum* sp from wild stock at Binuangen beach, Banten, Indonesia, which were harvested during low tide condition.

2.1. Samples preparation

The brown seaweed (*Sargassum* sp) was washed with fresh water followed by cleaning. The clean Brown seaweed (*Sargassum* sp) was washed with fresh water followed by cleaning. The clean material was soaked in boiled water with proportion of seaweed to water were varied i.e. 1:30 of 1 kg dry raw material, 1:20 of 3 and 10 kg dry raw material. Then 2% (w/v) 

| Supporting Material   | Unit | Raw Material Scale |
|-----------------------|------|--------------------|
|                       |      | 1 kg | 3 kg | 10 kg |
| Fresh water           | L    | 30.0 | 60.0 | 200.0 |
| Na₂CO₃ 2% (w/v)       | -    | 0.6  | 1.2  | 4.0   |
| Filter aid 3% (w/v)   | -    | 0.9  | 1.8  | 6.0   |
| HCl 1% (v/v)          | -    | 60.0 | 120.0| 400.0 |
| NaOCl                 | L    | 0.6  | 1.0  | 3.0   |
| CaCl₂                 | Kg   | 2.0  | 6.0  | 20.0  |
| IPA                   | L    | 3.0  | 9.0  | 30.0  |

The raw material soaked in boiled water with proportion of seaweed to water were varied i.e. 1:30 of 1 kg dry raw material, 1:20 of 3 and 10 kg dry raw material. Then 2% (w/v) Na₂CO₃ added and extraction is carried out on 60–70°C for 2 hour. The alginate solution obtained from extraction process was then acidified by adding 1% (v/v) HCl until the acidity of the solution reached a pH of 2 – 3 and then filtered by using vibrato screen. Bleaching process continued by adding 2% (v/v) NaOCl for 30 L extract and 1.5% (v/v) NaOCl for 60 and 200 L extract, slowly for 30 minutes.

The solution was converted into calcium alginate by adding 10% (w/v) CaCl₂ followed by smooth stirring for 10 minutes and finished when fiber is no longer formed. After pressed the fiber, conversion calcium alginate to sodium alginate conducted by adding Na₂CO₃ and mixed with the fiber using molen. The sodium alginate was then dehydrated using 10% IPA (for 30 L extract) and 15% IPA (for 60 and 200 L extract) followed by smooth stirring for 30 minutes until solid state of sodium alginate.
was formed. The solid state of the sodium alginate was dried at 50°C for 10 hours and then milled into powder using 100 mesh of sieve plate.

2.3. Water content analysis
Water content was analyzed using SNI 2534-1:2015 analysis procedure [11]. As much as 2 g sodium alginate powder were dried for 18-24 hours in the oven at 105°C, then cooled in a desiccator for 30 minutes and weighed after the samples reach room temperature (25°C). The value of water content is the percentage of the weight ratio of the water evaporated to the initial sample weight.

2.4. Viscosity analysis
Viscosity was measured based on Brookfiled viscometer manual book using Brookfiled LVT model viscometer. 7.5 grams of sodium alginate powder were dissolved in 500 ml of distilled water then stirred slowly at 80°C for 30 minutes. Analysis was conducted using a spindle number 2 with 60 rpm speed rotation and keeping the temperature of the solution stable. The stable number indicated by the tool was then multiplied by the conversion factor to obtain a viscosity value.

2.5. Data analysis
The experimental design used in this study was Completely Randomized Design with 3 replications. Data obtained were analyzed using MS-excel program.

3. Results and discussions
Extraction using 1 kg dry Sargassum sp. followed Murdinah et al[10]. Reproducibility of extraction method using this method will consume 4% NaOCl as bleaching agent which has unstable properties related with its characteristics as volatile acid solution during storage in room temperature. If NaOCl used in bleaching process can be reduced, the installation does not need to prepare a large chilling room for NaOCl storage. By keeping NaOCl in chilling room storage, proportion of NaOCl varied 4% and 2%. This solution has other characteristic i.e. oxidation agent. Therefore, viscosity of sodium alginate extracted from this experiment measure.

![Figure 1. Characteristics of sodium alginate produce from 1 kg Sargassum sp.](image)

Extraction using 3 kg dry Sargassum sp. followed modification Murdinah [10] method in bleaching process using 2% NaOCl. This bleaching solution storgaged in chilling room before used. Extraction using 3 kg raw material modified the 1 kg raw material method, i.e. ratio dry seaweed and fresh water
in extraction process and proportion of IPA. Ratio of raw material and fresh water used in extraction process usually 1:30, in pilot scale or higher scale it will consume a lot of fresh water. To minimize fresh water consumption, the method modified using ratio raw material and fresh water 1:20. Meanwhile, in original method dehydration process conducted by using IPA 10% (v/v). While modification will use IPA 15% (v/v). This modification conducted to maximize sodium alginate fiber production. Dehydration using 10% IPA produce a quite jelly fiber and it caused longer drying time. Dehydration process using 15% IPA will produce a unsoluble fiber of sodium alginate. Water content and viscosity are parameter that indicate the quality of sodium alginate produce by those modification.

![Figure 2. Characteristics of sodium alginate produce from 3 kg *Sargassum* sp.](image1)

Extraction using 10 kg dry *Sargassum* sp. followed modification Murdinah (2005) [10] method in bleaching process using ratio raw material to fresh water for extraction 1:20 and 15% IPA for water removal process from alginate fiber. NaOCl solution has oxidation properties, this solution has an ability to cut the alginate fiber and it will cause the viscosity of alginate lower. In this 10 kg scale production, the proportion of NaOCl modified from 2% to 1.5%.

![Figure 3. Characteristic of sodium alginate produced from 10 kg *Sargassum* sp.](image2)
4. Conclusion
Modification of the solvent carried out at each production scale, the proportion of bleaching material, sodium hypochlorite (NaOCl) on a scale of 1 kg of raw material 4% to 2% (v/v), the volume of water for extraction on a scale of 3 kg of raw material 30 to 20 times the weight of the raw material, the ratio of water binder, iso propyl alcohol (IPA) on a scale of 10 kg of raw material 8 to 3 times the weight of the raw material causes an increase in yield (17.97, 19.79, and 22.67%) and a decrease in viscosity (128.43, 55.03, and 40.27 cPs).

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