DOI: 10.21776/ub.jpacr.2019.008.01.420

J. Pure App. Chem. Res., **2019**, 8 (1), 23-30
20 January 2019

Extraction of Sodium Alginate from *Sargassum sp.* using Microwave-Assisted Extraction (MAE)

Ruslan,[1] Amran Amir,[2] and Agrippina Wiraningtyas[1*]

[1]Department of Chemical Education, Sekolah Tinggi Keguruan dan Ilmu Pengetahuan Bima, Jl. Tendean, Mande, Bima City, Nusa Tenggara Barat, 84019 Indonesia
[2]Department of Biology Education, Sekolah Tinggi Keguruan dan Ilmu Pengetahuan Bima, Jl. Tendean, Mande, Bima City, Nusa Tenggara Barat, 84019 Indonesia

* Corresponding email: agriwiraningtyas@gmail.com

Received 5 May 2018; Accepted 20 January 2019

ABSTRACT

The method used in alginate extraction has been using conventional heating. Lately, Microwave-Assisted Extraction (MAE) has been widely used to extract active compounds from natural ingredients. This study aims to extract sodium alginate from Sargassum sp. using MAE method. This research was conducted by determining the optimum power level and extraction time using a commercial microwave. Power level optimization is carried out at level 70; 80; 90 and 100, while extraction time is carried out for 15; 16; 17 and 18 minutes. The results showed that sodium alginate products were obtained at levels 70, 80, 90 and 100, respectively 0.5%; 37.13%; 23.36% and 1.2%. While sodium alginate products are obtained at variations in extraction time for 15; 16; 17 and 18 minutes were obtained respectively 27.4%; 37.13%; 26.1% and 25.76%. The characterization of sodium alginate products was carried out by analysis of water content, ash content, brightness, and viscosity in the sequence obtained by 14.43%; 14.63%; 78.62 and 95.00 cps. The results of the analysis of heavy metal content obtained that the product of sodium alginate extracted did not contain metal Pb and Hg. FTIR spectra results showed the presence of hydroxyl (-OH) functional groups at wave numbers 3448.7 cm^{-1} and 1620.21 cm^{-1}; C-H bond at 2931.8 cm^{-1}; alkene group at 2337.7 cm^{-1}; sodium (Na) bond at 1419.61 cm^{-1}; carbonyl group at 1095.57 cm^{-1} and carboxylic and ketone groups at 1033.85 cm^{-1}. Based on the results of the characterization proving that the product obtained is a sodium alginate compound.

Keywords: *Sargassum sp.* sodium alginate, physical and chemical properties, MAE method

INTRODUCTION

The wealth of diversity contained in the waters of Indonesia is quite high. One of the marine biological resources that are potential to be developed as it has a high economic value is seaweed *Sargassum* sp. which is a type of brown algae (*Phaeophyta*). Alginate is an ingredient that is contained by seaweeds of *Phaeophyceae* (brown algae). The industrial demand for sodium alginate every year is always an increase in both food and non-food industries. To meet these needs, Indonesia still relies on imported sodium alginate. The method used in the extraction of alginate, have been using conventional extraction methods. Several studies have been conducted on compounds of alginates with conventional methods [1-2]. The use of this method is very simple, but the drawback of this method is the extraction takes longer. Development of extraction methods to accelerate the extraction time is needed to reduce the cost of production.

Lately, the Microwave-Assisted Extraction (MAE) has been widely used to extract the active compounds in natural materials [3]. MAE method is an extraction that utilizes microwave radiation to speed up the extraction through solvent heating quickly and efficiently. The maximum microwave heating effect occurs at a frequency of 2450 MHz with an energy output of 600-700 watts. The heat of this microwave radiation can heat and evaporate water in the sample cells. So the pressure on the cell wall increases. As a result, the cell expands (swelling) and the pressure pushes the cell wall from within, stretching and breaking the cell. The destruction of plant cells facilitates the exit target compounds and extracted [4]. High temperatures of microwave radiation can hydrolyze the ether bonds on the cell wall components of plants, such as cellulose. In a short time, cellulose turns into a dissolved fraction. In addition, at elevated temperatures, it can increase dehydration of cellulose and decrease the mechanical strength of cellulose. As a result, the solvent is readily accessible to the target compound in the cell [5].

Based on some research results, the MAE method improves the efficiency and effectiveness of the extraction of active ingredients of various types of spices, herbs, and fruits [6]. The MAE method has advantages such as shorter required time, fewer solvents, reduced energy consumption, suitable for thermolabile components, yields higher extraction yields, greater accuracy, and precision [7]. Phenolic compounds have been isolated from seaweed *Eucheuma cottonii* using microwaves with optimum conditions at 60 °C with a 6 minute extraction time [8]. Extraction by MAE method utilizes microwave radiation to accelerate selective extraction through solvent heating in a fast and efficient manner. The advantages of extraction using the MAE method are reduced extraction time and less use of chemical solutions [9]. Previous research has been done to extract sodium alginate from seaweed *Sargassum* sp using conventional method compared with the MAE method [10]. This research will be conducted as sodium alginate extracted from seaweed *Sargassum sp*. using Microwave-Assisted Extraction (MAE) and characterization of its physical and chemical properties.

EXPERIMENT

Chemicals and instrumentation

Chemical and materials used in this study are seaweed *Sargassum* sp. taken from the Gulf of Bima, formalin (p.a.), hydrochloric acid (p.a.), sodium carbonate (p.a.), sodium hypochlorite (technical), ethanol (technical) and aquades.

Equipment used are glasses equipment, microwave (R-278 (S)), oven (Kirin), mixer, blender, pH universal, thermometer, Viscotester (Brookfield), color reader (Chromameter Konica Minolta CR-400), Atomic Absorption Spectroscopy (AAS) (Perkin Elmer) and Infrared Spectroscopy (FTIR Shimadzu 8201 PC).

Procedure reaction

Extraction Sodium Alginate

This procedure following the method of [10]. A total of 30 g of seaweed *Sargassum* sp. washed and dried. The dried seaweed soaked in a solution of 0.4% formalin for 6 hours and 1% hydrochloric acid solution for 1 hour and then washed with distilled water to pH neutral. Furthermore, seaweed cut added a solution of sodium carbonate 2% with a ratio of 1:30 (w/v). Subsequently extracted by Microwave at power level 70 for 15 minutes and then filtered. The filtrate obtained, was added 10% hydrochloric acid (until pH 2-3). Then do the bleaching with sodium hypochlorite diluted with water 1:1 to white. Then converted to sodium alginate by adding 20 g of sodium carbonate and stirred in a mixer. The resulting solution is then etched with ethanol to form sodium alginate fibers. Then filtering and pasta produced technical soaked in ethanol and dried in the sun for 12 hours until the moisture content of 12%. Furthermore, ground to a flour obtained sodium alginate.

Optimization of Power Level and Extraction Time

Optimization of power level and extraction time is taken to determine the effect of the level and timing with the MAE method. Power level optimization done on level 70; 80; 90 and 100 %. Time optimization of extraction was conducted at 15; 16; 17 and 18 minutes.

Characterization of Alginate

Characterization of sodium alginate was conducted to determine the physical and chemical properties of sodium alginate extraction results.

Analysis of Water Content

Analysis of water content is done by gravimetric methods of [11], by drying the sample at 105 ^{o}C alginate. The water content is determined based on the dry weight alginate that is a percentage of the dry weight of the initial weight.

Ash Content

Ash content analysis performed by gravimetric methods of [11], by burning the sample at 600 ^{o}C. Alginate ash content expressed as a percentage by weight of ash to the dry sample weight.

Analysis of Pb and Hg

Determination of Pb and Hg performed according to the method of [12], using the tool Atomic Absorption Spectrometry (AAS).

The yield

The yield of sodium alginate calculated following the [11] methods. The yield is the percentage by weight of alginate with an initial weight of seaweed.

Viscosity

Analysis of viscosity following the method of [12], which uses Viscotester. The samples used were 250 mL, which is obtained by making a 1% solution of alginate is heated at a temperature of 50 °C.

Brightness

Analysis of the brightness following the method of [12], which uses color reader. The brightness level samples, indicated by the value L. Value of L range between 0-100.

Structural Analysis

Structural analysis performed on the tool Infrared Spectrophotometer (FTIR) [13]. The sample is weighed as much as 1 mg mixed with KBr. Then the pellets are made of thin and inserted into the cell holder.

RESULT AND DISCUSSION

In this research extraction of sodium alginate was done from seaweed *Sargassum sp*. using Microwave-Assisted Extraction (MAE) method. The extraction process in this study was done by determining of optimum power level and extraction time. The results obtained were further characterized to determine physical and chemical properties.

Optimization of Power Level

Optimization of microwave power level is done to determine the optimum level with the highest yield. Data were obtained as listed in Table 1. Based on data in Table 1 shows that the highest yield of sodium alginate obtained at power level 80 by 37.13%, and decreased the yield on the 90 level that is equal to 23.36%. This is because the process of extracting the MAE method can increase the breakdown of cell walls in *Sargassum sp.* due to the microwave radiation beam.

Table 1. The yield of Sodium Alginate Optimization of Power Level

Power Level (%)	Yield (%)
70	0.5
80	37.13
90	23.36
100	1.2

MAE method involves heating using three times the energy conversion, namely the electrical energy into electromagnetic energy, then the kinetic energy and then into heat energy. The process of using microwave heating from the outside surface by conduction further into the sample so that the sample would be heated simultaneously. However, the higher the level resulting yield is lower because of alginate decomposed due to the structure of the higher thermal energy in the system.

Table 2. The yield of Sodium Alginate Time Optimization

Time (minute)	Yield (%)
15	27.4
16	37.13
17	26.1
18	25.76

Time Optimization

Further optimization is a variation of time to determine the most effective time to get the highest yield at 80 as an optimum level. The result of the time variation can be seen in Table 2. By the time variation showed that the highest yield (37.13%) was obtained in the extraction for 16 minutes, while the lowest yield (25.76%) in the 18th minute extraction method with Microwave-Assisted Extraction (MAE) is an effective method in comparison with maceration because it produces a higher yield of compounds, low temperature and a short time and improve the quality of the product [14]. Based on the standards of the Food Chemical Codex (FCC), the yield of sodium alginate produced is >18 % so that the sodium alginate extraction results already meets FCC standards.

Water Content

The water content of sodium alginate extraction results can be seen in Table 3. Based on observations indicate that the water content of sodium alginate extraction for 16 minutes resulted in low water content (14.43%) and the highest water content (18.74%) in the extraction for 18 minutes. The water content of the sodium alginate FCC is < 15% [2], while the sodium alginate from the extraction has an average water content of > 15%. This is due to the use of ethanol as sodium alginate extract washers absorb less water. Polarity ethanol has approached the polar of the water so it is more difficult to draw the water.

Table 3. Results of Water Content

Time (minute)	Water Content (%)
15	17.64
16	14.43
17	18.52
18	18.74

Ash Content

The ash content of sodium alginate extraction results is shown in Table 4. From the results obtained that the ash content of sodium alginate extraction results in the range of 14-21%. The ash content of sodium alginate extraction results meets quality standards alginate to finance exports with ash content of ≤ 24% [1].

Table 4. Results of Ash Content

Time (minute)	Ash Content (%)
15	16.37
16	14.63
17	16.13
18	21.08

Table 5. Results of Viscosity

Time (minute)	Viscosity (cps)
15	28.50
16	15.00
17	25.00
18	95.00

Viscosity

Test the viscosity of sodium alginate extraction results can be seen in Table 5. The data in show that the highest viscosity (95.00 cps) obtained in the extraction for 18 minutes while the lowest viscosity (15.00 cps) obtained in the extraction for 16 minutes. Sodium alginate has a low viscosity and this is because sodium alginate is easily ionized salt when reacted with water. Whereas in the old extraction has a high viscosity, this is due to hydroxyl and carboxyl high. Increased viscosity depends on the existence of guluronic acid and mannuronic acid are higher [1].

Brightness

The brightness of sodium alginate extraction results was measured using a tool Chromameter Konica Minolta CR-400. Sodium alginate produced generally beige or light yellow with Lightness value (L) the lowest (59.96) in the treatment period of 18 minutes and the highest value of L (78.60) in the treatment time of 16 minutes. Standard brightness or color of sodium alginate according to Farmakop Extras Indonesia is between white to yellow-brown [3], so the color of the sodium alginate extraction results in this study is in the standard range.

Identification of Pb and Hg

The secondary metabolite composed in *Sargassum sp* has previously reported correspond to their biological activity [15]. This paper analyzes toward the metals content especially Pb and Hg that was not reported previously. The results of the analysis of metal content Pb and Hg on sodium alginate performed by AAS method. The detection limit of the AAS instrument used is 0.01 ppm for Pb and 0.025 ppm Hg. Levels Pb set by the FCC is less than 10 ppm, while Hg levels as set by the FCC is less than 40 ppm [2]. Based on the analysis result of Pb and Hg metal content showed that extracted sodium alginate fulfilled the FCC standard.

Identification by Infrared Spectroscopy

Sodium alginate structural analysis conducted by testing the functional groups using infrared spectroscopy. Infrared spectra of sodium alginate extraction results as shown in Figure 1. The analysis of the spectra of sodium alginate extraction showed absorption at 3448.72 cm^{-1} region is stretching vibration absorption band of hydroxyl groups (O-H), it is confirmed from the absorption band at 1620.21 cm^{-1} region which is vibration buckling hydroxyl group (O-H). Weak absorptions in the area of 2931.80 and 617.22 cm^{-1} is an absorption band stretching vibration of C-H bond, the tape on the area 1419.61 cm^{-1} indicates the elements sodium (Na) isomer alginate, a weak band at 1095.57 cm^{-1} shows the group stretching vibration of C-O and C-C in pyranose ring and an absorption band at 1033.85 cm^{-1} region is stretching vibration carboxylic group (-COOH) and the ketone group (C-O-C). Band spectrum in the area of 948.98 cm^{-1} is the group stretching vibration of C-O uronic acid [13].

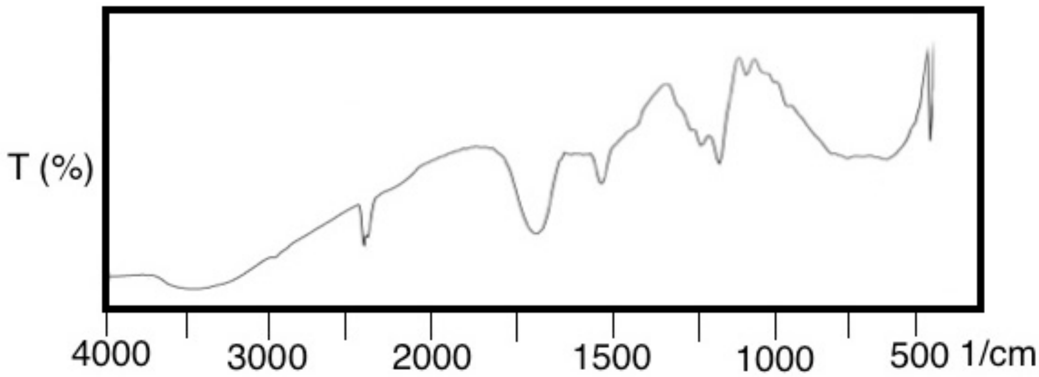

Figure 1. Infrared spectra of sodium alginate extraction results

Infrared Spectra of Sodium Alginate: Time Optimization

FTIR spectra of sodium alginate on time optimization as shown in Figure 2. From these spectra showed that the effect of the extraction time does not alter the pattern of the absorption band of sodium alginate, but differ in the magnitude of the intensity of the absorption band. In the treatment of extraction for 16 minutes showed a strong absorption band intensity or sharp at each functional group, but the longer the extraction time is for 17 and 18 minutes, the intensity of the absorption band is getting weaker. These data support the data of physical properties and chemical properties of sodium alginate extraction results. This proves that the optimum extraction conditions, namely in the treatment of sodium alginate extraction for 16 minutes.

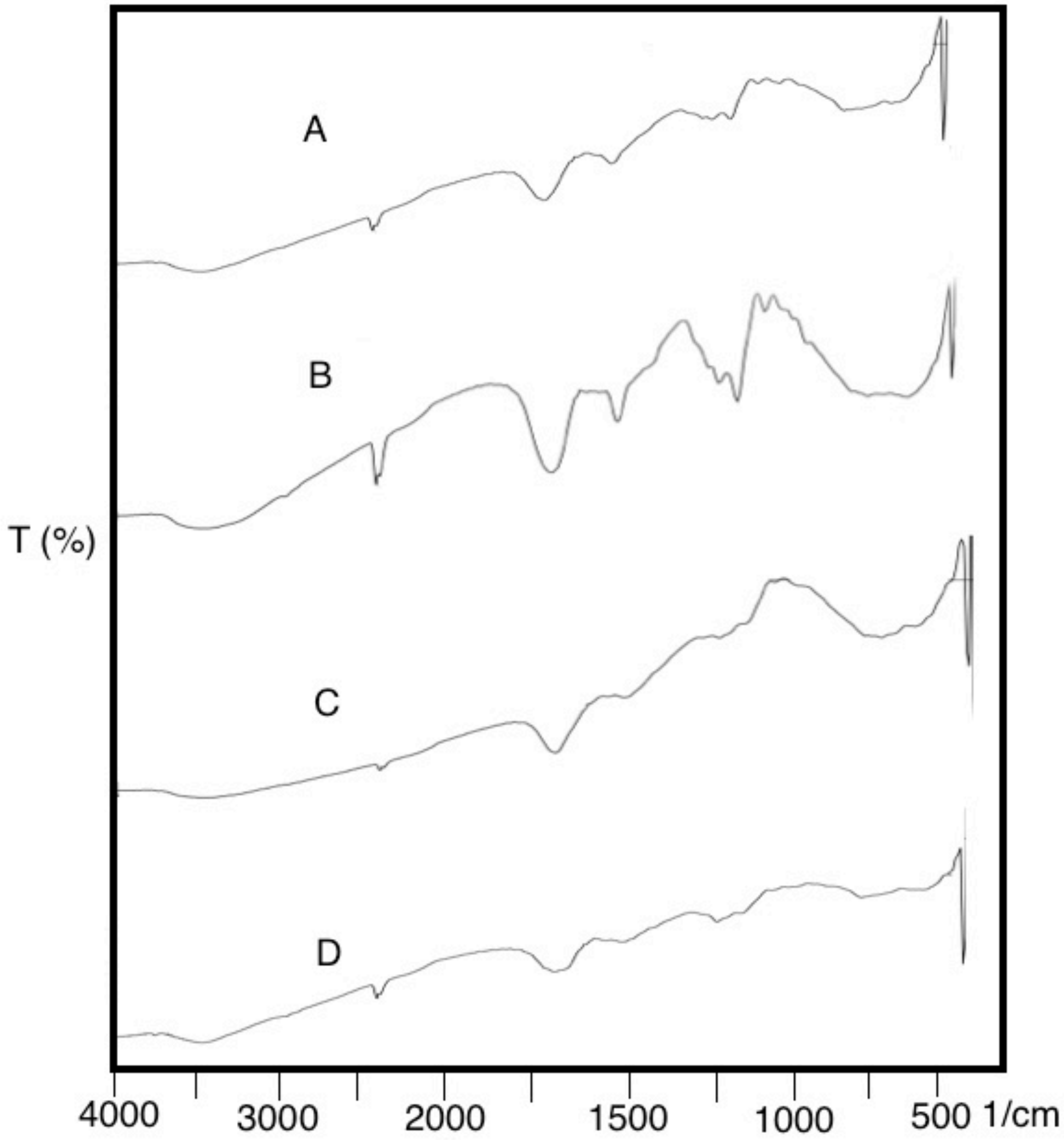

Figure 2. IR spectra of sodium alginate on time optimization: (a) 15, (b) 16 (c) 17 and (d) 18 minutes

CONCLUSION

Based on the results of this study concluded that sodium alginate extracted from seaweed *Sargassum sp*. by the MAE method can speed up the time of extraction and yield larger. The optimum condition MAE extraction method performed at 80 % microwave power level with a yield of 37.13%, 14.43% moisture content, an ash content of 14.63%, 78.62 brightness in treatment for 16 minutes while the viscosity of 95.00 cps at treatment for 18 minutes. In the structural analysis obtained spectral absorption band indicating a hydroxyl group (O-H), carboxyl (COOH), carbonyl (C = O), carbon ketone (C-O-C) and the bonding of sodium (Na) thus proving that the sample extraction is sodium alginate.

ACKNOWLEDGMENT

The authors thank the Directorate of Research and Community Service Directorate General for Strengthening Research and Development of the Ministry of Research, Technology and Higher Education of the Republic of Indonesia, which has funded this research through Competitive Research Grant.

REFERENCES

[1] Amir, H., Subaryono, Pranoto, Y., Tazwir dan Ustadi, *Agritech*, **2012**, 32(1), 1-8.
[2] Mushollaeni, W., *Afr. J. Food* Sci., **2011**, 5(6), 349–352.
[3] Silva, M., Gomes, F., Oliveira, F., Morais, S., Matos, C.D.. *Int. J. Chem., Nucl., Matter. Metallurgical Eng.,* **2015**, 9(1), 30-33.
[4] Jain, T., Jain, V., Pandey, R., Vyas, A., & Shukla, S*., Asian J. Res. Chem*. **2009**. 2(1), 19-25.
[5] Mandal, V., Mohan, Y., & Hemalatha, S. *Pharmacogn Rev*. **2007**. 1(1). 7-18.
[6] Ghallisa, K.N., Nugroho, W.A., Hendrawan, Y., *Jurnal Bioproses Komoditas Tropis*, **2014**, 2 (1), 72-78.
[7] Handayani, D., Mun'im, A., Ranti, A.S., *Trad. Med. J.*, **2014**, 19 (1), 29-35.
[8] Kartikasari, D., Wardhani, D.H., Prasetyaningrum, A., *Jurnal Teknik Kimia*, **2013**, 19(3), 38-43.
[9] Rafiee, Z. S. M., Jafari, M., Alami, Khomeiri, *J. Anim. Plant Sci.*, **2011**, 21(4), 738-745.
[10] Amir, A., Wiraningtyas, A., Ruslan, dan Annafi, N, *Chempublish Journal*, **2016**, 1(2), 7-13.
[11] AOAC, *Official Methods of Analysis of the Association of Official Analytical Chemistry*, 15th Edition, **1990**, Washington, 456-579.
[12] James, CS., *Analytical Chemistry of Foods*, **1995**, Blackie Academic and Profesional, London, 80-95.
[13] Silverstein, R.M. Webster, F.X. Kiemle, D.J., *Spectrometric Identification of Organic Compounds*, 8th edition, **2005**, Jhon Wiley & Sons Inc., New York, 79-80.
[14] Fertah, M., Belfkira, A., Dahmane, E.M., Taourirte, M., *Arab. J. Chem.*, **2017**, 10 (Supplement 2), S3707-S3714.
[15] Fauziah, F., Aulani'am, A., Mahdi, C., *J. Pure App. Chem. Res.*, **2013**, 2(3), 102-107.