The purity and viscosity of sodium alginate extracted from *Sargassum* brown seaweed species as a basic ingredient in dental alginate impression material

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**Abstract.** Sodium alginate is one of the basic ingredients of alginate impression materials. Experimental sodium alginate was made by using an immersion method in acid to extract *Sargassum* brown seaweed species. The sodium alginate powder was then tested for its purity and viscosity. The experimental sodium alginate had a purity determined by high-performance liquid chromatography equivalent to that of a standard sodium alginate powder (SIGMA A2158). The Brookfield viscosity test showed that the viscosity of the experimental sodium alginate was too low (45.3 mPas) and unsuitable for use as a dental alginate impression material basic ingredient.

1. Introduction

Alginate materials are commonly used to make impressions in dentistry. Currently, many dentists use alginate impression material because it can provide accurate impression results [1]. Alginate materials are used to make study models, for treatment evaluations, and to make impressions before making a crown, bridge, or removable prosthesis [1]. Alginate impression material undergoes a phase change from the solid phase to the gel phase via a chemical reaction, which occurs when sodium alginate is mixed with water. Therefore, when the gelation is complete, the alginate cannot return to the solid phase and the impression material is called an irreversible hydrocolloid [1,2,3].

The manufacture of alginate impression materials require several basic ingredients, including sodium alginate, calcium sulfate, sodium sulfate, diatomic soil, flavorings, dyes, and disinfectants [2]. In manufacturing impression materials, the working time, setting time, viscosity, compatibility with gypsum, dimensional stability, and filler compatibility must be considered [4,5,6]. The basic material of sodium alginate impression material can be obtained from *Sargassum* sp. brown seaweed [7].

*Sargassum* sp. grows at a depth of 0.5–10 m under water with a heavy stream of water. This brown algae requires a greater amount of light than does other algae. The length of the main thallus of the algae *Sargassum* sp. is 1–3 m and has a bubble in each branch called a bladder, which is useful to support the thallus branches floating up to the surface to receive sunlight [8].

Alginate is one group of polysaccharides formed from brown algae cell walls. Alginate is more commonly known as a copolymer of L-guluronic acid and D-mannuronic acid. Alginate has three kinds of structures: α-1,4-L-guluronate; β-1, 4-D-mannuronate; and heteropolysaccharide, which is a
form of α-1,4-L-guluronate and β-1, 4-D-mannuronate acid [9]. Sodium alginate (or algin) can be obtained from brown grass Sargassum sp., which is extracted in an acidic environment, and is one of the organic macromolecule electrolytic types of brown seaweed species with the chemical formula (C₆H₇NaO₆)ₙ [9].

When brown seaweed Sargassum sp. is used for alginite impression materials, it is important that the extracted sodium alginate has the proper purity and sufficient viscosity for the impression material to provide an appropriate contour [2]. This purity can be tested by using high-performance liquid chromatography (HPLC).

HPLC requires a solvent called a mobile phase that carries dissolved compounds through the HPLC column, which contains a solid stationary phase [10]. An HPLC system includes a mobile phase reservoir(s), pump(s), injector, column (containing the stationary phase), detector, mobile phase discharge container, detector (to detect compounds of interest), and a data acquisition system that can generate chromatograms showing compound peaks versus time and can measure the peak areas, heights, and retention times [11,12,13]. A sample of the test solution is injected into the mobile phase, which carries the sample through the column containing the stationary phase. The compounds dissolved in the test solution are separated according to their relative affinities for the stationary and mobile phases. Compounds with a greater affinity for the stationary phase take longer (have longer retention times) to reach the detector, which records each compound as a peak, with the height and area being proportional to the concentration of the compound [10].

A viscosity test is performed on sodium alginate from brown seaweed Sargassum sp. Viscosity is the flow resistance of a liquid (fluid). A Brookfield viscometer can be used to measure viscosity and has fluid and spindle viscosity readers. The sample is first heated and then placed into the viscometer. A spindle selection is performed according to the viscosity of the sample. The speed of the viscometer is set and the needle that rotates is adjusted until it is stable, which indicates the viscosity of the fluid [14].

The purpose of this study was to determine the purity and viscosity of brown seaweed Sargassum sp. obtained from Binuangen, Serang, Banten, as dental alginate impression material.

2. Methods
In this study, sodium alginate was extracted from brown seaweed Sargassum sp. obtained from Binuangen, Serang, Banten, by washing the dried brown seaweed thoroughly and then soaking it in 1% HCl at a rate as high as 1:30 (w/v) for 1 day. Then, the seaweed material was boiled with added 2% Na₂CO₃ 1:30 (w/v) at 60–70 °C for 60 min, ground, and boiled at 60–70 °C for 60 min. Blanching was performed by using NaOCl 0.25% for 30 min and then adding HCl 10% for alginate acid formation up to a pH of 2.8–3.2. Then, the material was washed with the acid alginate until the pH was neutral. The alginic acid was converted to sodium alginate with 10% NaOH addition until the pH reached 7–8. The sodium alginate was extracted into isopropyl alcohol for 30 min and then dried in the sun continued by milling into flour.

After the sodium alginate was obtained, its purity was determined by using HPLC. The first step was the preparation of 10,000 ppm sodium alginate extraction test sample and SIGMA A2158 10,000 ppm sodium alginate reference standard. The solution was prepared by weighing 250 mg of each powder, mixing it with 25 mL water, and then stirring the mixture until the solids dissolved. Then, 20 μL of the solution was injected into the HPLC instrument for analysis. The HPLC conditions were as follows: carbohydrate column with water as the mobile phase, 1 mL/min flow rate, 40 °C column temperature, and UV detection at a 300 nm wavelength.

The viscosity test was performed by using a Brookfield viscometer. The sodium alginate extract test sample and SIGMA A2158 sodium alginate reference standard were each prepared as solutions (7.5 g in 500 g of water). The water and powder were mixed in a measuring cup and covered with aluminum foil and then heated by using an AM4 Multiple Heating Magnetic Stirrer at 60 °C for 30 min. The viscosity testing was performed by using a Brookfield Model LV viscometer with a spindle number of 2 for the sodium alginate extract solution and a spindle number of 1 for the standard
SIGMA A2158 sodium alginate powder at 60 rpm. The test was performed three times for each sample.

3. Results
In this study, the purities of the SIGMA A2158 *Sargassum* sp. alginate standard solution, the extraction test sample, and a mixed solution of the standard and test sample were tested. The results are presented in Table 1. The major peaks in all three preparations had nearly identical retention times of approximately 5.4 and 10.4 min. Other peaks at different times and smaller areas indicate the presence of impurities in the solvent or in the sodium alginate powder.

| Specimen | Concentration (ppm) | Retention time (min) | Area width (nm) |
|----------|---------------------|----------------------|-----------------|
| Natrium Alginate *Sargassum sp.* | 10.000 | 5.48 * | 109.017 |
| SIGMA A2158 Standard Alginate | 10.000 | 5.39 * | 98.979 |
| Mixture of Natrium Alginate *Sargassum sp.* | 10.000 | 5.41 * | 105.413 |
| Standard Alginate | 10.000 | 8.37 | 2.803 |

Table 1 HPLC analysis results for the SIGMA A2158 alginate standard, Natrium Alginate *Sargassum sp.* extraction test sample, and a mixture of the standard and extraction test sample

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Further study was conducted to test the viscosity of the sodium alginate powder, and the results are presented in Table 2. The sodium alginate sample extracted from *Sargassum* sp. had a viscosity of 45.3 mPas, and the SIGMA A 2158 alginate standard had a viscosity of 20 mPas.

| Specimens | Speed (rpm) | Spindel | Viscosity (mPas) |
|-----------|-------------|---------|-----------------|
| Sodium Alginate extracted from *Sargassum sp.* | 60 | No. 2 | 45.3 |
| Alginate Standard “SIGMA A2158” | 60 | No. 1 | 20 |

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*Annotation*: Peak which shows at the same retentive time for natrium alginate with the biggest surface area
4. Discussion
The HPLC purity test results showed that the purities of the sodium alginate powder extracted from Sargassum sp. and of the SIGMA A2158 standard alginate solution were similar according to peak retention times and areas. The HPLC purity test results showed that the purity of the sodium alginate extracted from Sargassum sp. sodium alginate was similar to the purity of the SIGMA A2158 sodium alginate that was used as a standard.

A solution containing a mixture of the standard and test sample was prepared to see if the peaks observed in the single solutions remained at the same retention times in the mixed solution. This is called a spiked solution, which can show if there is any effect of the sample preparation matrix on the retention times (or peak areas) of the main compounds [15]. The HPLC test results for the mixture showed that the main peaks (largest peak areas) remained at approximately 5.4 and 10.4 min, which confirmed that the peaks were associated with sodium alginate. These results indicated that the extraction of the seaweed Sargassum sp. successfully isolated the sodium alginate from the raw seaweed [15].

In the viscosity test, the sodium alginate extracted from Sargassum sp. had a viscosity of 45.3 mPas and the SIGMA A2158 standard alginate powder had a viscosity of 20 mPas. According to Rashid, the viscosity of sodium alginate can be divided into three categories: (1) low viscosity, <240 mPas; (2) medium viscosity, 240–3500 mPas; and (3) high viscosity, >3500 mPas [9]. This classification indicates that both the alginate extracted from Sargassum sp. obtained from the Binuangen area, Serang, Banten, and the SIGMA A2158 standard sodium alginate powder were of low viscosity.

The low-viscosity properties of sodium alginate may affect the viscosity of a prepared alginate impression material. For the manufacture of alginate impression materials, the viscosity of sodium alginate must be ≥300 mPas (moderate viscosity) [16]. Viscosity is related to the fluidity of a liquid. Low-viscosity sodium alginate has a high flow rate, and high-viscosity sodium alginate has a low flow rate that will affect the setting time [17].

The viscosity of sodium alginate can also be influenced by aquatic conditions, pH, salinity, light, depth of the water from which the seaweed is extracted, and nutrients (As, Pb, Hg, ash, and water). Another factor that affects viscosity is the morphology of brown seaweed because the shape of the thallus of each type of brown seaweed is different. A short thallus will produce low-viscosity sodium alginate; a long thallus will produce a high-viscosity sodium alginate [18,19]. Viscosity is also affected by the weight of the alginate molecule. The molecular weight determines whether the viscosity of sodium alginate is low or high. A low molecular weight will have a low viscosity, whereas a high molecular weight will produce a high viscosity [20].

Table 1 presents the viscosity test results for the solution of sodium alginate extracted from Sargassum sp., which has a viscosity of 45.3 mPas indicating that the molecular weight of the alginate sodium powder is also low. On the basis of research conducted by GC Corporation, sodium alginate powder extracted from Sargassum sp. is necessary to increase the molecular weight to achieve a suitable viscosity in the preparation of alginate impression materials [16]. The increased molecular weight of the sodium alginate powder from Sargassum sp. will increase the viscosity, which will produce a setting time in accordance with the ISO1563 1978 standard.

M. Darmawan showed that a change to 0.1% KOH for 60 min in the immersion part of the extraction method of Sargassum sp. may increase the viscosity of sodium alginate extracted from Sargassum sp. relative to that achieved by using 0.33% HCL [19]. In the present study, the immersion of Sargassum sp. was performed in 1% HCL, which might have given the low viscosity of sodium alginate extracted from Sargassum sp.

This study showed that extraction of sodium alginate from Sargassum sp. yielding high purity material but with low viscosity or lower purity material but with higher viscosity gives material that is unsuitable for making alginate dental impressions. Therefore, pure sodium alginate extracted from Sargassum sp. with high viscosity is needed to make an alginate impression material that has a suitable consistency and setting time. Increasing the viscosity can be achieved by soaking the extracted Sargassum sp. in KOH.
5. Conclusion
This study showed that the purity of sodium alginate extracted from *Sargassum* sp. was similar to that of SIGMA A2158 sodium alginate standard material. However, the obtained high purity sodium alginate from *Sargassum* sp. had a viscosity that was too low for use as alginate impression material.

6. References
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