Preparation of experimental dental alginate impression material from *Sargassum* spp. seaweed extract based on its setting time

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Abstract. Alginate impression material is one of the most widely used impression materials in dentistry because it is easy to use, economical, and exhibits excellent impression results. Indonesia’s coastal waters are rich in brown seaweed, one of which is *Sargassum* brown seaweed, which can be processed into alginate and eventually used in dentistry. Thus, this preliminary, laboratory-based experimental study aims to prepare experimental dental alginate impression material from *Sargassum* spp. seaweed extract obtained from Binuangeun (Banten, Indonesia) based on its setting time tested according to ANSI/ADA Specification for Alginate Impression Materials no. 18: 1983. After extracting *Sargassum* spp. seaweed, the alginate powder was mixed with its water/powder ratio obtained from the combination of alginate composition and tested for its final setting time. The obtained impression material from *Sargassum* spp. seaweed was in the form of sodium alginate powder extract, which was light brown, odorless, and shaped like fine sand. This study suggests that the preparation of experimental impression materials from seaweed extract obtained from *Sargassum* spp. has a setting time of 3 min corroborating ANSI/ADA Specs no. 18: 1983, signifying the potential to be used with various other additives to produce Indonesia’s alginate impression materials.

1. Introduction

Dental alginate impression material is an ideal and most prevalent material for recording or producing a replica of the form and relation of teeth and oral tissues [1]. Impressions can be either created of one tooth, all teeth, or the edentulous jaw. The alginate impression is known to provide a negative reproduction of the oral tissue, and positive reproduction can be obtained by filling the mold with dental stone or other removable casting material after the hardening of the gypsum model [1]. Typically, the impression material is used to assess the orthodontic treatment, diagnostic purpose, and the process of making restorations or prostheses [2]. Apparently, the alginate impression material is one of the most widely used impression materials in dentistry because it is easy to use, economical, and exhibits excellent impression results [2,3].

In Indonesia, the dental alginate impression material is widely used because of its affordable price and almost accurate impression result. However, because Indonesia imports the alginate impression material, its availability is highly dependent on importers. Typically, alginate impression materials are available in the powder form that also contains other substances, including sodium alginate, calcium sulfate, sodium phosphate, diatomic soil, and other substances [4]. The primary component of a dental
alginate impression material is formed by water-soluble alginates, such as sodium, potassium, or triethanolamine alginate. The combination of water-soluble alginate and water quickly results in the formation of the sol phase. The higher the molecular weight of the alginate mixture, the thicker the sol formed [2]. The mixture of powder with water and the product formed are then used to evaluate the mouth tissue.

One of the compositions, sodium alginate (a sodium salt contained in alginic acid), can be obtained from the extraction of brown seaweed [5]. All types of brown seaweed contain alginate; however, only a few species are extracted for commercial production because of its more favorable alginate content, such as *Macrocystis pyrifera* (America), *Ascophyllum nodosum* (Canada), *Laminaria hyperboreae*, and *Laminaria digitata* (France). Indonesia’s coastal waters are rich in brown seaweed, one of which is *Sargassum* brown seaweed, which can be processed into alginate and eventually be used in dentistry [6]. Alginate, commonly abbreviated as algin, is contained in the cell walls of brown seaweed, contributing to the seaweed’s versatility [7]. Brown seaweed (*Phaeophyceae*) contains sodium alginate or alginate (Na-Alginate). Alginic acid comprises 1-mannuronic acid and 1-guluronic acid and is a sap membrane (membrane mucilage or natural gummy), whereas alginate is a salt form of alginic acid. The most vital polysaccharides in brown seaweed are alginic acids and their derivatives, such as fucoidan, funoran, and laminar, which constitute the components of the cell wall [8].

Thus, this preliminary, laboratory-based experimental study aims to prepare experimental dental alginate impression material from *Sargassum* spp. seaweed extract based on its final setting time and the water/powder ratio.

### 2. Methods

This experimental laboratory research was divided into two procedures, as follows: (a) making sodium alginate powder from seaweed extract obtained from *Sargassum* spp.; (b) making experimental alginate impression materials with various powder compositions over time of setting. To obtain the seaweed extract from *Sargassum* spp. seaweed, dried seaweed was soaked in 1% HCl (1:30 b/v) and extracted with 2% Na₂CO₃ (1:30) at 70 °C for 60 min. Next, re-grinding and re-extracting were performed at 60–70 °C for 60 min, followed by filtering and then purging with NaOCl (0.25% of the filtrate volume) for 30 min. 10% HCl was added to reach a pH of 2.8–3.2, by which alginic acid was formed, followed by washing the alginate acid until neutral. After that, the conversion to sodium alginate was performed by adding 10% NaOH to pH 7–8, followed by separating sodium alginate in IPA (1:2 v/v) and stirring before leaving for 30 min. Finally, the product was dried in the sun and floured to obtain sodium alginate powder [9].

In the procedure of making the experimental alginate impression material over the setting time, the composition was divided into powder composition I (5 g of sodium alginate powder and different water quantities, 20–27 mL), powder composition II (addition of sodium alginate powder composition with calcium sulfate powder), and composition of powder III (mixture of sodium alginate powder, calcium sulfate powder, and sodium phosphate powder with water). Only 5 g of sodium alginate powder was used in this experiment because of its limited availability. The preparation of an alginate impression material with powder composition I intended to attain the best ratio of the sodium alginate powder mixture with water. Meanwhile, the preparation of the powder composition II intended to evaluate the setting time and the dough results after the powder was mixed with water.

In powder composition III, sodium alginate powder calcium sulfate powder, and sodium phosphate powder were mixed with water. The test was conducted by gradually adding a significant amount of sodium phosphate powder to obtain appropriate consistency based on the best ratio of the alginate impression material from powder composition I. The preparation of the alginate impression material with powder composition III intended to assess the mixture after the reaction. Finally, the setting time of the powder III (final) composition was recorded.
3. Results

The extraction process from Sargassum spp. seaweed yielded light brown colored, odorless, sodium alginate powder, shaped like fine sand with a particle size of 80 mesh. Table 1 summarizes the results of preparing the alginate impression material with powder composition I. The best ratio of the sodium alginate powder mixture of Sargassum spp. seaweed extraction results with the criteria of the commercial impression material (GC Aroma Fine Plus) was obtained at the ratio 24 mL of water and 5 g of sodium alginate powder (water ratio and powder I) with a setting time of 3-min. Table 2 summarizes the results of preparing the alginate impression material with powder composition II. The ratio closest to the best ratio was obtained with the addition of 0.1 g of calcium sulfate powder with a setting time of 3-min. Table 3 summarizes the results of preparing the alginate impression material with powder composition III. Of note, the usage of water more than 24 mL resulted in a non-creamy mixture because it was more dilute and the mixture could not harden. Thus, the ratio of 24 mL water and 5 g of powder was the best ratio in the preparation of alginate impression materials of powder compositions II and III to obtain the results of alginate dough used to record the jaw.

| Criteria       | Results                              |
|----------------|--------------------------------------|
|                | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 |
| Lump           | Yes | Yes | Yes | Yes | No | No | No | No |
| Dissolved      | Yes | Yes | Yes | Yes | Yes | Yes | Yes | Yes |
| Stirring       | Hard | Hard | Hard | Easy | Easy | Easy | Easy | Easy |
| Consistency    | Creamy | Creamy | Creamy | Creamy | Creamy | Non-creamy | Non-creamy | Non-creamy |
| Flow           | Bad | Bad | Good | Good | Good | Good | Good | Good |
| Torn           | Yes | Yes | No | No | No | No | No | No |
| Elasticity     | Elastic | Elastic | Elastic | Elastic | Elastic | Plastic | Plastic | Plastic |
| Setting Time   | 1 min | 1 min | 2 min | 2 min | 3 min | 3 min | Unset | Unset |

1 = 20 mL Water/5 g NaAlg
2 = 21 mL Water/5 g NaAlg
3 = 22 mL Water/5 g NaAlg
4 = 23 mL Water/5 g NaAlg
5 = 24 mL Water/5 g NaAlg
6 = 25 mL Water/5 g NaAlg
7 = 26 mL Water/5 g NaAlg
8 = 27 mL Water/5 g NaAlg

| Criteria       | Results                              |
|----------------|--------------------------------------|
|                | 24 mL H2O+5 g NaAg/0.1 g CaSO4 | 24 mL H2O+5 g NaAg/0.2 g CaSO4 | 24 mL H2O+5 g NaAg/0.3 g CaSO4 | 24 mL H2O+5 g NaAg/0.5 g CaSO4 |
| Lump           | No | No | No | No |
| Dissolved      | Yes | Yes | Yes | Yes |
| Stirring       | Easy | Easy | Easy | Easy |
| Consistency    | Creamy | Creamy | Non-creamy | Non-creamy |
| Flow           | Good | Good | Good | Good |
| Torn           | No | No | No | No |
| Elasticity     | Elastic | Elastic | Elastic | Elastic |
| Setting Time   | 3 min | 3 min | 5 min | 5 min |
Results of preparing the alginate impression material with powder composition III.

| Criteria       | 24 mL H$_2$O+5 g NaAg + 0.1 g CaSO$_4$/0.1 g Na$_3$PO$_4$ | 24 mL H$_2$O+5 g NaAg + 0.1 g CaSO$_4$/0.2 g Na$_3$PO$_4$ | 24 mL H$_2$O+5 g NaAg + 0.1 g CaSO$_4$/0.3 g Na$_3$PO$_4$ | 24 mL H$_2$O+5 g NaAg + 0.1 g CaSO$_4$/0.5 g Na$_3$PO$_4$ |
|----------------|----------------------------------------------------------|----------------------------------------------------------|----------------------------------------------------------|----------------------------------------------------------|
| Consistency    | Creamy                                                   | Creamy                                                   | Creamy                                                   | Non-creamy                                               |
| Flow           | Good                                                     | Good                                                     | Not Good                                                 | Not Good                                                 |
| Stirring       | Easy                                                     | Hard                                                     | Hard                                                     | Hard                                                     |
| Dissolved      | Yes                                                      | Yes                                                      | Yes                                                      | Yes                                                      |
| Lump           | No                                                       | Yes                                                      | Yes                                                      | Yes                                                      |
| Torn           | No                                                       | Yes                                                      | Yes                                                      | Yes                                                      |
| Elasticity     | Elastic                                                  | Elastic                                                  | Elastic                                                  | Elastic                                                  |
| Time           | 3 min                                                    | 3 min                                                    | 2 min                                                    | 2 min                                                    |

Table 3. Results of preparing the alginate impression material with powder composition III.

4. Discussion

This experimental study resulted in the preparation of the impression material from Sargassum spp. seaweed in the form of sodium alginate powder extract, which was light brown, odorless, and shaped like fine sand. The extracted sodium alginate powder was then used as the basic material of the experimental alginate impression material with the additions of the compositions comprising calcium sulfate as a gelling agent and sodium phosphate as a retarder, accounting for prolonged setting time [1].

In this study, the best ratio was obtained with 5 g of sodium alginate powder and 24 mL of water with 3-min setting time, in accordance with the setting time recommended by ANSI/ADA Specification No. 18: 1983, which states that the normal setting time of an alginate impression material should be 3–4 min and 30 s. The combination of calcium salt and sodium alginate with water produces a water-insoluble elastic gel known as calcium alginate [10]. The chemical reaction is as follows:

\[ n\text{Na}_2\text{Ag} + n\text{CaSO}_4 \rightarrow n\text{Na}_2\text{SO}_4 + n\text{Ca}_n\text{Ag} \]

Sodium Alginate       Calcium Sulfate       Sodium Sulfate       Calcium Alginate

In addition, the best ratio with powder composition II was obtained by the addition of 0.1 g of calcium sulfate powder with a 3-min setting time. However, the preparation of the alginate impression material II reveals that higher the amount of calcium sulfate powder added, more dilute the mixture becomes (non-creamy) with a prolonged setting time, which inversely correlates with the theory explaining that the addition of calcium sulfate forms a quick bond between calcium sulfate and sodium alginate to form calcium alginate in the form of gel [2]. A study anticipated that mixed results should be obtained with a creamy consistency and faster setting time after adding more calcium sulfate [11].

It is observed that sodium phosphate in the dental alginate impression powder acted as a retarder or setting time-inhibiting agent. In addition, the calcium sulfate ion, which should directly bind to sodium alginate, first reacted with phosphate ions of sodium phosphate to form insoluble calcium phosphate. Previously, calcium phosphate has been proven to be formed more than calcium alginate because of its low solubility [1]. Another study reported that after the depletion of the phosphate ion, calcium reacts with water-soluble alginate to form insoluble calcium alginate, which combined with water, forms an irreversible calcium alginate gel [8].

Based on theories and the results from Table 3, the addition of 0.1 g of sodium phosphate powder with a setting time of 3 min was the best ratio in accordance with the setting time proposed by ANSI/ADA Specification No. 18: 1983. However, the obtained results inversely correlated with the theory because the addition of a higher amount of sodium phosphate powder shortened the setting time.
Perhaps, longer setting time should be obtained by adding more sodium phosphate. Although the cause of this nonconformity remains unclear, it supposedly occurs because of the unsuitable powder of natural seaweed extraction by mixing sodium phosphate powder and calcium sulfate in the form of finished material (manufactured).

This study has some limitations. First, the ratio of 24 mL of water:5.2 g of the powder obtained in this study comprised only three basic compositions of an alginate impression material, 5 g of sodium alginate, 0.1 g of calcium sulfate, and 0.1 g of sodium phosphate. Thus, to obtain an experimental alginate impression material by better extracting Sargassum spp. seaweed, it is imperative to add the composition of other substances, such as diatomic soil as filler particles, potassium titanium fluoride as accelerator reaction, potassium sulfate to provide better surface quality, a coloring agent, and a flavoring agent, in commercial dental alginate impression materials [1].

5. Conclusion

This study suggests that the preparation of experimental impression materials from seaweed extract obtained from Sargassum spp. has a setting time of 3 min corroborating ANSI/ADA Specs no. 18: 1983. Hence, the basic composition of the experimental alginate impression material has the potential to develop and be used with various other additives to produce Indonesia’s alginate impression materials.

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