Making of Alginate Dental Impression Material from *Sargassum duplicatum*

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Abstract This study was aimed to prepare dental impression material from *Sargassum duplicatum* taken from Binuangeun, Banten. The setting time was tested according to ANSI/ADA Specification for Alginate Impression Materials 1983. After extraction, obtained the alginate powder then mixed its water/powder ratio from the combination of alginate composition and tested final setting time. Variations of the formulations used different calcium sulfate (0,1 g; 0,2 g; and 0,3 g). Product quality parameters are lumps, solubility, consistency, flow, tearing, setting time. The alginate yield obtained from *Sargassum duplicatum* by 31 % with characteristic gel strength by 816 (gr/cm²); viscosity 238 mPas (2%; room temperature). The highest ratio is the best ratio by adding calcium sulfate powder of 0.1 gram with a setting time of 3 minutes.

1 Introduction

Since 1940, alginate dental impression materials has been introduced [1]. Since then they have been used extensively in dentistry as one of the group of so-called 'elastic' impression materials [2]. Alginate impression material is one of the most widely used impression materials in the field of dentistry because it is easy to use, economical, and provides good impression results. Another reason of using alginate include the low cost, a better tolerability on the part of the patient, the ease of manipulation, the short time needed for execution [3][4]. In Indonesia, dental alginate impression materials are widely used because the price is quite affordable and the printouts are quite accurate. However, the type of alginate impression material is still imported from abroad so the availability of this material is very dependent on the importer. Alginate impression materials are generally available in powder form containing a number of compositions, including sodium alginate, calcium sulfate, sodium phosphate, diatomic soil, and other substances [5]. The powder is then mixed with water and produces a mixture that is used to print mouth tissue. One of its main compositions, sodium

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alginate which is a sodium salt contained in alginic acid, can be obtained from the extraction of brown seaweed [6]. In this regard, it is necessary to carry out preliminary research to make alginate impression material that commonly found in Indonesian waters. In this study, various compositions used will be reviewed against the hardening time and the ratio of powdered water according to ANSI / ADA No. Specifications. 18: 1983 for dental alginate impression materials [7]. Potassium salt and sodium salt from alginic acid have properties that make alginate can be used as a dental impression material. Alginic acid, which is made from marine plants, is a high molecular weight block copolymer of anhydro- β -D-manuronic acid and anhydro- β -D-guluronic acid. The nature of the alginate raw material mainly depends on the degree of polymerization and the ratio of guluronate and mannuronate blocks in polymer molecules.

Fig. 1. Structure calcium alginate [2]

The manuronate area is stretchy and flat, while the guluronate region has low flexibility. In addition, generally guluronate blocks bind to Ca^{+2} . Therefore, alginate which is rich in guluronate forms a strong and brittle gel, while that rich in mannuronate form gels that are weaker and more form a weak and more elastic gel [2]. The ability of alginates to form gels is mainly related to the proportion of L-guluronate blocks. The chemical structure of alginate can be seen in Figure 1[2]. In a mixture of alginate printing materials, the powder contains calcium sulfate dihydrate, soluble alginate, and sodium phosphate. Calcium ions from calcium sulfate dihydrate react with phosphate ions from sodium phosphate and phosphophosphate to form insoluble calcium phosphate. Calcium phosphate will be more formed than calcium alginate because it has a lower solubility, so sodium phosphate is called a retarder because it provides working time for alginate mixtures [8]. The aim of research was study the ratio of water with alginate powder and calcium sulfate toward setting time.

2 Materials and methods

2.1 Materials

Alginate powder from seaweed extract obtained from *Sargassum duplicatum*, Binuangeun, Banten.

2.2 Extraction of alginate

To obtain alginate powder, dried seaweed was soaked in 1% HCl (1:30 b/v) and extracted with 2% Na₂CO₃ (1:30) at 60-70 °C for 60 min. Next, the seaweed ground and re-extracting at 60-70 °C for 60 min, followed by filtering and then added with NaOCl (0.25% of the filtrate volume) for 30 min. Then 10% HCl was added to reach a pH of 2.8–3.2 to separate alginic acid from filtrate, followed by washing the alginic 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, and leaving for 30 min. Finally, the product was dried in the sun and grinded to obtained sodium alginate powder [9]. The characterization of alginate (yield, gel strength, viscosity, moisture content, pH) and as comparison used alginate commercial; making experimental alginate impression materials with various calcium sulfate compositions over time of setting.

2.3 Making the alginate impression material based on setting time

The first, sodium alginate and different water quantities (20-27 mL) are mixed interval 1 mL. 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. After that mixed alginate powder, sodium phosphate with various calcium sulfate (0,1 g; 0,2 g and 0,3 g) were mixed with water. Finally, the setting time of the powder composition was recorded. The criteria assessment include: lump, dissolved, stirring, torn, flow, elastic, and setting time.

3 Results and discussion

3.1 Characterization of alginate powder

The results of the quality of alginate flour from extraction of *Sargassum duplicatum* seaweed obtained as shown in Table 1. Based on the results of the analysis, the alginate from *S. duplicatum* have higher viscosity and gel strength compared to commercial alginates. High viscosity and gel strength were indicated that this alginate contain high glucuronate content compared to manuronates. This is because guluronate compounds more easily interact with cations that have a valence of 2, thus increasing gel strength and viscosity [2]. Conversely, the higher mannuronic acid produces a weaker and more elastic gel, with better freeze-thaw stability behavior [3] [5]. For dental impression materials, it is expected that alginate was easily binding with calcium and has a good elasticity (based on the requirements according to ANSI/ADA Specification for Alginate Impression Materials no. 18: 1983).

Table 1. Characteristic of alginate powder

Parameters	Result of analysis	
	Alginate from <i>Sargassum duplicatum</i>	Standard alginate (Sigma A2158)
Yield (%)	31	-
Colour powder (visual)	Light brown	beige
Moisture content (%)	16.95	12.4
Whiteness degree (%)	44.1	-
Viscosity at room temperature (2,0%) in mPas	238	105
Viscosity at 75 °C (2,0%) in mPas	84	40
Gel strength (1.5%) + CaCO ₃ 20 mM; GDL 30 mM after 2 hours (g/cm ²)	816	420

3.2 Making of alginate dental impression material

From the results of mixing water as much as 20 ml; 21 ml; 22 ml; 23 ml; 24 ml; 25 ml; 26 ml; 27 ml, with the same amount of sodium alginate powder as 5 grams, the following results are presented in Table 2. From this studied the best ratio of water and sodium is 24 mL/5 grams alginate. The use of more than 24 ml of water produces a consistency of the mixture that is non-creamy because it is increasingly runny, and the mixture no harden.

Table 2. Results of making the alginate impression material with different alginate ratio

Criteria	Water alginate ratio							
	20 mL/ 5 g Sodium alginate	21 mL/ 5 g Sodium alginate	22 mL/ 5 g Sodium alginate	23 mL/ 5 g Sodium alginate	24 mL/ 5 g Sodium alginate	25 mL/ 5 g Sodium alginate	26mL/ 5 g Sodium alginate	27 mL/ 5 g Sodium alginate
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	Elastic	No elastic	No elastic
Setting time	1 min	1 min	2 min	2 min	3 min	3 min	No hard	No hard

Furthermore, the formulation of making alginate impression material with a variety of calcium sulfate was conducted. The mixing results obtained as shown in Table 3. The addition of calcium sulfate that meets the standards according to ANSI / ADA Specification for Alginate Impression Materials no. 18: 1983 based on setting time is 0.1 gr with a 3 minutes setting time.

Table 3. Characteristic of alginate impression material at different calcium sulfate

Criteria	24 mL H ₂ O/5 g Sodium alginate +0,1 gr CaSO ₄	24 mL H ₂ O/5 g Sodium alginate +0,2 gr CaSO ₄	24 mL H ₂ O/5 g Sodium alginate +0,3 gr CaSO ₄
Lump	No	No	No
Dissolved	yes	Yes	Yes
Stirring	Easy	Easy	Easy
Consistency	<i>creamy</i>	<i>Creamy</i>	<i>Non creamy</i>
Flow	Good	Good	Good
Torn	No	No	No
Elasticity	Elastic	Elastic	Elastic
Setting time	3 min	3 min	5 min

However, the obtained results inversely correlated with the theory because the addition of a higher amount of sodium phosphate powder shortened the setting time. The combination of calcium salt and sodium alginate with water produces a water-insoluble elastic gel known as calcium alginate [11]. The chemical reaction is as follows [7]:

Addition of calcium sulfate to alginate forms a calcium alginate compound and is a by-product of sodium sulfate. Calcium alginate has soft gel properties, water insoluble, and gelatinous. The results of the reactions can be seen in Figure 2.

Fig. 2. Mixed alginate and calcium sulfate

To obtain a better time setting than commercial standards it is necessary to add other ingredients like titanium fluoride as acceleration reaction. Several materials were potassium sulfate can using to produce smooth surface quality, MgO to higher tear energies, pigment, flavor as like as product of alginate dental impression commercial [9]. Impression materials

must have ability to reproduce a true measured value and they should be dimensionally stable across time [16]. By this research given information that *Sargassum duplicatum* potential has potential to be further developed dental alginate impression material product original from Indonesia.

4 Conclusion

Based on the findings of this study, the best ratio of water with alginate powder by adding calcium sulfate powder of 0.1 gram with a setting time of 3 minutes. The overall results obtained by the formula of dental alginate molds consisting of a water ratio of 24 mL, 5 g sodium alginate and 0.1 g of calcium sulfate, obtained a time setting of 3 minutes.

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