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Original article

The analysis of accelerator concentration effect on setting time and morphology surface impression materials made of brown algae *Sargassum* sp☆

Bahruddin Thalib[a,*], Nurlindah Hamrun[b], Mohammad Dharma Utama[a], Edy Machmud[a], Siti Arpa[a], Asmawati[b], Rafikah Hasyim[b], Alqarama Mahardhika Thalib[c]

[a] *Prosthodontic Department, Faculty of Dentistry, Hasanuddin University, Makassar, Indonesia*
[b] *Oral Biology Department, Faculty of Dentistry, Hasanuddin University, Makassar, Indonesia*
[c] *Dental Material Science & Technology Department, Faculty of Dentistry, Hasanuddin University, Makassar, Indonesia*

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ABSTRACT

Objective: To analyze the accelerator concentration on alginate impression material setting time made of brown algae *Sargassum* sp.
Method: This study was an experimental laboratory using samples of impression material made of brown algae *Sargassum* sp. Measurement of setting time using stopwatch and morphological surface analysis using Scanning Electron Magnetic (SEM).
Result: The average setting time for the fastest calcium sulfate accelerator is 3.11 min (17 g) and the longest is 6.13 min (15 g). As for the fastest potassium titanium fluoride accelerator material the timeline setting is 3.81 min (4 g) and the longest is 5.73 min (2 g). SEM analysis showed that alginate printing impression materials with accelerator materials of calcium sulfate 17 g and potassium titanium fluoride 4 g showed the smaller, finer denser and more homogenous surface morphology.
Conclusion: Impression material made of *Sargassum* sp. with accelerator calcium sulfate 17 g and potassium titanium fluoride 4 g has a better setting time and surface morphology.

Introduction

Alginate is used as a preliminary impression to make an individual tray, study models that help in the making of treatment plans, removable partial denture impression, orthodontic models, and restoration treatment plans.[1–8] Alginate impression materials are available in powder form containing several components, one of the main components of which is potassium alginate which is soluble in water.[9,10]

Potassium alginate can be obtained from the extraction of brown algae (*Phaeophyceae*). There are several brown algae that can produce alginate, one of which is *Sargassum* sp.[11] Brown algae grow wild and their potential has not been utilized in industrial fields, especially in the manufacture of impression materials in the field of dentistry.[12] Other compositions are diatomaceous earth and zinc oxide, accelerator material in the form of calcium sulfate and potassium titanium fluoride and retarder material in the form of trisodium phosphate.

The addition of accelerator materials is important because it can regulate the time needed for material manipulation, applications in the oral cavity until the material becomes stiff or reaches a certain elasticity known as setting time. The setting time is related to the time used till material reaches the final thickening or hardening status. One of the tools for calculating the setting time by using a stopwatch.[10] Clinically, the normal time setting required by hydrocolloid irreversible impression materials is 3–4.5 min.[9,10] It is important to pay attention on how much accelerator material is added to alginate impression material because it can affect the morphological quality of impression surface.

Accurate dental impression is strongly influenced by the setting time and surface smoothness of impression using irreversible hydrocolloid alginate impression material. The objective of this study was to analyze how much the concentration of accelerator material addition on alginate impression material made of brown algae *Sargassum* species, to produce the right setting time and good morphological surface to obtain high-accuracy impression.

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* Corresponding author.
E-mail addresses: bathalib64@yahoo.com, pmc@agri.unhas.ac.id (B. Thalib).

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Material and method

This study was an experimental laboratory using brown algae *Sargassum* sp., 5% HCl, 4% Na_2CO_3, 12% NaOCl, 10% Ca_2CO_3, isopropanol (IPA), calcium sulfate, trisodium sulfate, diatomaceous earth, potassium titanium fluoride, zinc oxide, aquades, and waterone.

Extraction of potassium alginate Sargassum *sp.*

Brown algae *Sargassum* sp. obtained from the coastal waters of Putondo-Punaga, Takalar District, South Sulawesi Province, Indonesia was extracted to produce potassium alginate.

Five kg dried brown algae was soaked in HCl 1% solution for 1 h, then cleaned with distilled water, mixed with 4% Na_2CO_3 solution afterward then heated at 60 °C for 2 h, then diluted with distilled water and blended, then filtered. The filter is filtered with 12% NaOCl solution, 5% HCl was added (pH 2–3) to form a lump of alginate acid foam, then filtered again, washed and added with 10% Ca_2CO_3 to reach pH 7. 95% isopropanol was added on alginate acid with a ratio of 1:2 to be converted to potassium alginate and then frozen for up to 12 h. Then dried into the freezer dryer, blended and sifted with 200 mess to become potassium alginate powder.

Preparation of impression material made of brown algae Sargassum *sp.*

Dental impression materials was made by mixing all prepared ingredients (potassium alginate extracted from *Sargassum* sp. Brown algae, diatomaceous earth, zinc oxide calcium sulfate, trisodium phosphate and potassium titanium fluoride) at a certain percentage using mortar and pestle. After all components of the material were mixed evenly followed by sifting using a 200 mesh, a total of 45 samples were prepared with a 15 sample calcium sulfate accelerator formulation, 15 samples of titanium fluoride potassium and 15 control samples (*Hygedent*), then each formula was divided into 3 groups with 5 times repeated measurements. Formula samples can be seen in the following table:

Setting time measurement

A total of 4 g of impression material for each formula were mixed with distilled water for 30 seconds at 23 °C, then poured in cylindrical molded containers with a diameter of 30 mm and height of 16 mm. The setting time is measured/recorded using a stopwatch, starting at the time of mixing the material until the impression material was no longer attached to the indicator tool.

Data were obtained, collected and processed with ANOVA test to determine setting time differences in each group of alginate impression material formulations made of brown algae *Sargassum* sp. LSD Post-Hoc test were performed to determine the difference in the setting time value between each group, compared to the control impression material. Chi square test was used in observing surface morphology. The confidence level was 95% or the value of $p = 0.05$.

Impression surface morphology

The SEM tool used in this study was "Inspect S50". Data were obtained, collected and processed with ANOVA test to determine setting time differences in each group of alginate impression material formulations made of brown algae *Sargassum* sp. LSD Post-Hoc test was performed to determine the difference in the setting time value between each group, compared to the control impression material. Chi-square test was used in observing surface morphology. The confidence level was 95% or the value of $p = 0.05$.

Result

Setting time

The average setting time on each impression material and comparing it with the control impression material can be seen on the following table.

Table 1 showed that the impression material with the fastest setting time is the Ca3 formula which is 3.112 min then followed by the C2 formula 4.152 min and the longest setting time is the formula Ca1 with 6.136 min. The control impression material has an average setting time of 3.024 min.

Table 2 showed that the impression material with the fastest setting time is T3 formulation with 3.814 min, followed by a T2 formulation 4.342 min, and the longest one is a T1 formula with 5.736 min. The control material has an average setting time of 3.024 min.

The average setting time of Ca3 formula almost has the same time with the control material, 3.112: 3.024 min and statistical tests also showed differences though are not significant, while for the formula Ca1 and Ca2 showed significant differences (Table 3).

The average setting time of T3 formula has the same time with control material. This result was also reinforced by the results of statistical tests that show no significant difference, while for formulas T2 and T1 the results showed significant setting time differences (Table 4).

A very small difference between C3, T3 and control material formulas, thus no significant difference with those two formulas and control (Table 5).

Table 1
Average setting time (min) of several calcium sulfate formulation in alginate impression materials made of brown algae *Sargassum* sp. and standard alginate as control materials.

No.	Group	*N*	Minimum	Maximum	Mean ± SD
1	Impression Material Ca1	5	6.00	6.25	6.136 ± 0.096
2	Impression Material Ca2	5	3.43	5.30	4.152 ± 0.753
3	Impression Material Ca3	5	3.07	3.19	3.112 ± 0.048
4	Control Material	5	3.01	3.03	3.024 ± 0.009

Table 2
Average setting time (min) of several potassium titanium fluoride formulations in alginate impression materials made of brown algae *Sargassum* sp. and standard alginate as control materials.

No.	Group	*N*	Minimum	Maximum	Mean ± SD
1	Impression material T1	5	5.41	6.12	5.736 ± 0.311
2	Impression material T2	5	3.47	5.17	4.342 ± 0.826
3	Impression material T3	5	3.10	5.30	3.814 ± 0.906
4	Control material	5	3.01	3.03	3.024 ± 0.009

Table 3
Analysis result of post-hoc LSD formula C1–C3.

No.	Group	Mean differences	*p*
1	Ca1 vs. control	3.112	0.000
2	Ca2 vs. control	1.128	0.000
3	Ca3 vs. control	0.088	0.719

One-way ANOVA. Post-hoc LSD: Ca 1 vs. control, $p < 0.05$; Ca2 vs. control, $p < 0.05$; Ca3 vs. control, $p > 0.05$

Table 4
The analysis result of post-hoc LSD formula T1–T3.

No.	Group	Mean differences	*p*
1	T1 vs. control	2.712	0.000
2	T2 vs. control	1.318	0.000
3	T3 vs. control	0.790	0.066

One-way ANOVA. Post-hoc LSD: T1 vs. control, $p < 0.05$; T2 vs. control, $p < 0.05$; T3 vs. control, $p > 0.05$.

Table 5
Result analysis of post-hoc LSD Ca3, T3 and control.

No.	Group	Mean	*p*
1	Ca3 vs. T3	0.702	0.056
2	Ca3 vs. control	0.088	0.795
3	T3 vs. control	0.790	0.066

One-way Anova test. Post-hoc LSD: Ca3 vs. control, $p > 0.05$; Ca3 vs. control, $p > 0.05$; T3 vs. control, $p > 0.05$.

Surface morphology of alginate impression material

Fig. 1 showed the results of surface morphology by SEM analysis at 20,000 magnification. Ca1 alginate material showed larger and sharper particles, while the surface on Ca3 formula showed the smoothest surface, the smallest particle size, density and homogenous. This mixture profile is a good profile for impression material because it will easily follow the contours of the teeth and tissues contained in the oral cavity.

Fig. 2 showed the results of surface morphology examination by SEM analysis at 20,000 times magnification. T1 alginate impression material showed a profile that resembles a honeycomb which is a characteristic of alginate-based impression material. Characteristics of hollow materials like this are not good for an impression because it showed that the material was not mixed and blends well. T3 impression material showed a continuous and tight profile compared to T1 and T2 materials.

Discussion

Several studies has been carried out regarding brown algae from Indonesian Makassar waters, the results showed several physical properties that resemble standard alginate impression materials.[13] Setting time is the time used to manipulate the material into certain elasticity. The material usually used to adjust the setting time is an accelerator in the form of calcium sulfate and potassium titanium fluoride, while the retarder can be in the form of trisodium phosphate.[9,10]

The results of setting time examination showed that the increased percentage of calcium sulfate and potassium titanium fluoride accelerators led to a faster setting time of alginate impression materials. This showed that the higher the percentage of the accelerator, the faster the formation of alginate gel occurs. The impression material with formulation of calcium sulfate 17 g (C3) accelerator has the fastest setting time of 3.112 min, while the 4 g (T3) titanium fluoride potassium accelerator has the fastest setting time with 3.814 min, while the control setting is 3.024 min. Based on the recommendations of the American Dental Association (ADA) no. 18 and ISO 1563 there are 2 types of setting time for alginate, namely fast set with a setting time of 1–2 min and normal set with a setting time of around 3–4.5 min. Thus, the results of this study fall into the category of alginate with a normal set or regular set.[14–16]

The setting time must be sufficient to give practitioners time when mixing ingredients, placing the material into the tray, and into the patient's mouth, giving comfort to patients not to open their mouths for too long. The setting time can also be influenced by several factors including water powder ratio, water temperature used, room temperature, stirring technique, mixing time and composition of alginate.[17,18]

The results of one-way ANOVA LSD post-hoc test showed that the comparison between alginate impression material *Sargassum* sp. C3 formula and T3 formula with control material showed no significant differences ($p > 0.05$) which means that the setting time of the formula corresponds to the standard impression material that circulates in the market.

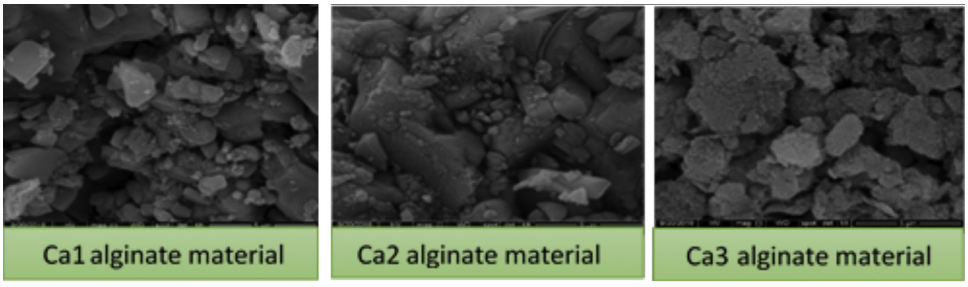

Fig. 1. SEM analysis result on several calcium sulfate formulation of alginate material made of brown algae *Sargassum* sp.

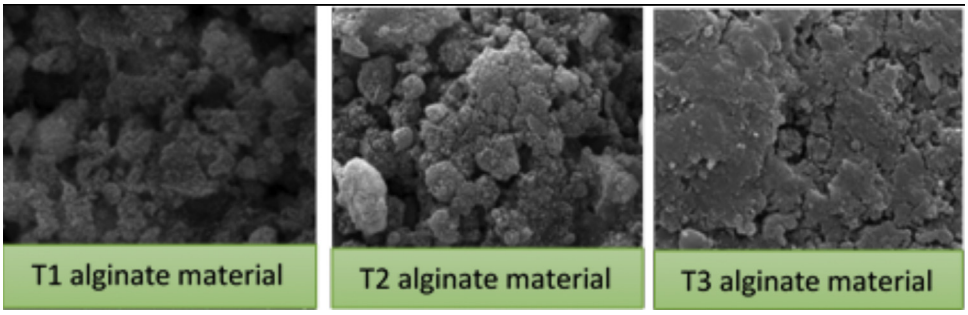

Fig. 2. Honey comb appearance on SEM analysis result in several potassium titanium fluoride.

In mixing powder and water, soles and alginates are formed, salt, calcium, and phosphate begin to dissolve. The gel formation is blocked by trisodium phosphate which reacts with calcium sulfate to produce calcium phosphate deposits. Calcium phosphate is formed earlier than calcium alginate, this is due to lower levels of calcium phosphate solubility compared to calcium alginate. Trisodium phosphate found in alginate, as a retarder will affect the working time when mixing water with alginate material. After the phosphate ion has reacted, the calcium ion will react with soluble alginate (water-soluble alginate) to form calcium alginate. The replacement of the monovalent sodium group with divalent calcium produces cross bonds in the alginate chains and the conversion of material from the sol form to the gel. When a setting reaction occurs, and the degree of crosslinking increases, the gel gives off elastic properties.[9,10]

By comparing the alginate impression formula Ca3 and T3, it can be concluded that the brown algae impression material with calcium sulfate 17 g (Ca3) accelerator has the setting time that is closest to the control impression material time setting, namely 3.112: 3.024 min and Ca3 impression formula has the best morphological surface because it has smaller particle size, less porous and homogeneous so that it shortens the setting time of impression material compared to T3 formula material. Impression materials with smaller size particle and smaller porous are very recommended to be used as teeth and tissue impression in oral cavity, because they can replicate dental tissue and its surrounding areas with a high degree of accuracy.

Conclusion

Sargassum sp. brown algae impression material with accelerator composition of calcium sulfate 17 g and potassium titanium fluoride 4 g has a better setting time and surface morphology compared to impression materials with other formulas.

Conflict of interest

The authors declare no conflict of interest.

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