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Elastic Recovery Properties of Alginate Impression Materials Based on Red Algae *Kappaphycus alvarezii*

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ORIGINAL ARTICLE

Elastic Recovery Properties of Alginate Impression Materials Based on Red Algae *Kappaphycus alvarezii*

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ABSTRACT

Alginate impression materials in dentistry are used to form details of the oral cavity organs. The alginate content in the impression material functions to form a gel. One source of alginate is algae. **Objective:** This study aims to determine the recovery elasticity and setting time of alginate impression materials made from red algae *K. alvarezii*. **Methods:** The sodium alginate in the composition of this alginate impression material was extracted from red algae *K. alvarezii*. The alginate impression material was tested for setting time and recovery elasticity and compared with commercial impression material (Hygident[®]). **Results:** The elastic recovery of red algae sodium alginate impression materials was 97.43% which was not significantly different from the commercial impression materials (98.42%). These results showed that this alginate impression material has the same properties as commercial impression materials. In the setting time test, the red algal alginate impression material was much longer than the commercial impression material, 1.39 m and 3.30 m respectively. **Conclusion:** It was concluded that the alginate impression material from the red algae *K. alvarezii* had a long setting time. At the same time, the recovery of elasticity did not differ from that of commercially available alginate impression materials.

Key words: alginate, dental impression, elastic recovery, hydrogel, *K. alvarezii*

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INTRODUCTION

Alginate impression material is widely used in dentistry and can imprint accurate replicas of the hard and soft tissues of the oral cavity. Alginate impression material has several advantages, such as its ability to print replicas of the shape of teeth and soft tissues around the oral cavity, patient comfort, and easy mixing and modification with simple equipment.¹ Alginate impression materials are primarily composed of algin, commonly found in the form of alginic acid or alginate. Sodium alginate can be derived from natural materials such as algae.^{2,3} Brown algae species are rich in sodium alginate polysaccharides and are a source of sodium alginate, which is widely used in commercial alginate impression materials.⁴ The red algae species *K. alvarezii* also contains alginic acid polysaccharides, so it has the potential to be utilized as an alginate

impression material.⁵ Previous research shows that red algae-based dental alginate has dimensional stability no different from that of factory-made dental alginate,⁶ and its viscosity is relatively high by standards, 33,200cP.⁷ Once molded in the oral cavity, alginate impression materials tend to be unstable due to the influence of syneresis and imbibition. Therefore, alginate impression materials must conform to the American National Standard Institution/American Dental Association (ANSI/ADA) Specification No. 18-1992. Mechanical properties must be met, including strain, elasticity recovery, and tensile strength. These properties determine the success or failure of the impression material; for example, the alginate impression material must remain unchanged when the impression is removed from the oral cavity after passing through the undercut region.⁸ The impression material should have a high elastic limit. The material

should not change or be able to return to its original shape after pressure and force are removed. This event is called elastic recovery.⁹

Alginate with a higher manuronate content makes the material structure more elastic, while a higher guluronate content makes the material more rigid.¹⁰ The elasticity of impression materials can affect dimensional stability through the physical properties of impression materials through viscosity. The lower the viscosity of impression material, the higher the elasticity will be, and the lower the permanent deformation will be, so that the elastic recovery will be high.¹¹ The viscosity of the *K. alvarezii* alginate impression material is lower than of commercial alginate impression materials, so it has the potential to produce more elastic impression materials.⁷ The use of red algal polysaccharides as an alternative dental impression material must meet the requirements based on ANSI/ADA specifications, one of which is good elastic recovery.

Elastic recovery is the percentage of the ability of an impression material to return to its original dimensions after removal from the oral cavity. Based on ANSI/ADA specifications, the ideal elastic recovery for alginate impression materials is greater than 95%. The elasticity of the impression material influences the elastic recovery. Impression materials must have good elasticity properties to print the undercut area.¹² The elasticity of impression materials can be influenced by several factors, such as the content of the alginate structure, the viscosity of the impression material, the setting time of the impression material, and the amount of pressure and time when the material is under pressure.^{2,13} The alginate structure contains of manuronate and guluronate monomers, which can affect the elasticity of the alginate impression material.

Based on the above description, it is necessary to test the elastic recovery of alginate impression materials based on red algae *K. alvarezii* which is expected to provide good elastic recovery according to ANSI/ADA specifications so that it can be used as an alternative to alginate impression materials in dentistry.

METHODS

The type of research used is a laboratory experiment with a post-test-only control group design. Two research group used in this study. The first group used a commercial impression material (Hygedent®), and the second group used an impression material whose sodium alginate composition was derived from red algae extract. A total of four samples used in each group. Both groups were tested for elastic recovery and setting time.

The sodium alginic acid extraction process

Red algae (purchased from farmers) were washed

with running water and then soaked in 0.1% potassium hydroxide (KOH) (Merck) for 1 hour. The algae were rewashed and dried in an oven at 60^o C for 96 hours until the water content reached <15%. The dried algae was ground to powder using a blender. One hundred grams of algae powder was soaked in 1% hydrogen chloride (HCl) (Merck) solution (1:30 w/v) for approximately 1 hour, then washed until the pH became neutral. Extraction was performed using 2% sodium carbonate (Na₂CO₃) (Merck) solution (1:30 w/v) for 2 hours and shaken in a water bath shaker at a temperature of 60-70^o C. It was then, filtered to obtain the filtrate. The filtrate was purified with 10% NaOCl (Merck), 4% by volume, for 30 minutes until it was ivory yellow. The filtrate was titrated with 10% HCl until the pH reached 2.8-3.2. It was allowed to stand until an alginic acid precipitate was obtained. The precipitate was filtered and Na₂CO₃ solution was added until the pH became neutral to obtain sodium alginate. Isopropyl alcohol (Merck) solution (1:2 v/v) was added for 30 minutes and then filtered. The resulting sodium alginate was dried in an oven at 60^oC and ground using a blender.¹⁴

Identification of *K. alvarezii* sodium alginate components

The sodium alginate obtained was tested with a Fourier Transform InfraRed (FTIR) spectrophotometer (Cary 630 FTIR spectrometer, USA) to show the absorption peak contained in the extract. Sodium alginate samples were powdered, placed on a plate, and analyzed. The absorption peak of the FTIR results was analysis to determine the functional groups of sodium alginate.

Preparation of alginate dental impression

All the necessary ingredients to prepare alginate impression materials were mixed using a mortar, including calcium sulfate (Merck) 14%, potassium sulfate (Merck) 10%, diatomaceous earth (Asia Lab) (filler) 50%, trisodium phosphate or trisodium phosphate (Merck) 2%, Hydroxy Propyl Methyl Cellulose (HMPC) (Merck) 6% and 18% sodium alginate from red algae. After mixing, the alginate impression material powder was obtained by blender and filtered with a 150-mesh filter.¹⁵

Sample preparation

The manipulation of alginate impression material in both groups was done conventionally (by hand stirring at the same time and speed) or by hand mixing. The ratio of water to powder ratio was 5 grams: 11.5 ml of water.

Setting time test

Prior to performing the elastic recovery test, a setting time test was carried out. The setting time test was formed. The setting time was performed using an acrylic rod 10 cm long and 6 mm in diameter. The impression material was mixed with water in a rubber container, stirred manually with a spatula, and poured

into a ring-shaped mold with a diameter of 20 mm and a height of 10 mm. The setting time was measured by inserting the acrylic rod into the mold and lifting it every 10 seconds until the material no longer adhered. The setting time was calculated from the beginning of mixing the impression material with water until the mixture no longer adhered to the measuring instrument. The setting time was calculated in seconds from the beginning of mixing the impression material with water until the mixture no longer adhered to the measuring instrument.¹⁶

Elastic recovery test

Elastic recovery measurements were performed using an elastic recovery testing device in accordance with ANSI/ADA Specification no. 18. Elastic recovery testing began with the preparation of a mixture of impression materials. The dough was poured into the fixation ring, pressed with a press, covered with a glass plate, clamped with a C-clamp, and placed in a 37° C water bath. Alginate samples were placed in an elastic recovery tester (Mitutoyo Europe GmbH, Neuss, Germany). The upper surface of the sample was covered with a small glass plate. The test was performed in several time steps (seconds) starting at t+45 seconds. The indicator button was lowered until it touched the glass plate on the sample, and then at t+55 seconds, the reading on the indicator needle was taken. The value read is recorded as A. The needle was raised again at t+60 seconds. The sample was pressed to a height of 16 mm within 1 second, held for 5 seconds ± 0.5 seconds, and then released to its original position. At t+90 seconds, the indicator button was lowered until it touched the glass plate above the sample. At t+100 seconds, the indicator needle is read, and the value was recorded as B. Where is the measurement result of the setting time.¹⁷ The elastic recovery is calculated in percent (%) using the following formula (ANSI/ADA No. 18, 1992).

$$t = 100 \times \left(\frac{1 - (A - B)}{20} \right)$$

Description:

- t = Setting time measure result
- A = Initial value read on the indicator dial
- B = Final value read on the indicator dial
- 20 = mold length (in millimeters)

Data analysis

Data were analyzed using SPSS computer software (IBM SPSS Statistic 25) to perform statistical tests. Data were tested for normality using the Shapiro-Wilk test, then the homogeneity was tested using Levene's test and then the T-test was performed.

RESULTS

Figure 1 is the result of FTIR analysis in the form of functional group absorption peaks showing the compounds in the red algae extract (*K. alvarezii*). The

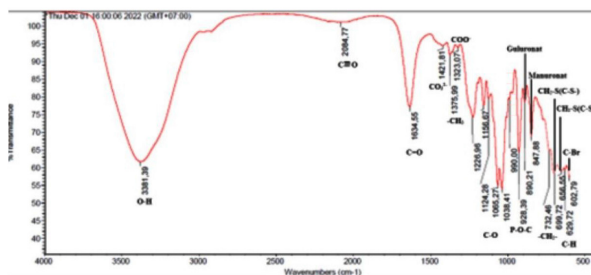


Figure 1. FT-IR spectra of sodium alginate from red algae (*K. alvarezii*).

Table 1. Average setting time measurement results.

Sample	Setting time	
	Commercial	<i>K. alvarezii</i>
1	1.38 ± 0.035	3.01 ± 0.53
2	1.41 ± 0.044	3.26 ± 0.52
3	1.38 ± 0.010	3.36 ± 0.34
4	1.39 ± 0.071	3.57 ± 0.60
Average	1.39 ± 0.038	3.30 ± 0.49

Table 2. Average elastic recovery measurement result.

Sample	Elastic recovery test (%)	
	Commercial	<i>K. alvarezii</i>
1	98.26 ± 0.20	98.56 ± 0.25
2	98.43 ± 0.30	96.43 ± 0.24
3	97.91 ± 0.22	97.93 ± 0.31
4	99.08 ± 0.20	96.80 ± 0.26
Average	98.42 ± 0.23	97.43 ± 0.26

specific absorption peak for hydroxyl groups (O-H) was at wave number 3381.39 cm-1, for cyanide groups (C=O) at wave number 2084.77 cm-1, for carbonyl groups (C=O) at wave number 1634.55 cm-1, for carbonate ion (CO32-) at wave number 1421.81cm-1, for methyl group (-CH3) at wave number 1375.99 cm-1, for carboxylate ion (COO-) at wave number 1323.23cm-1, for carboxyl group (C-O) at wave number (1226.96 cm-1 -1038.41 cm-1), for aliphatic phosphate group (P-O-C) at wave number 990 cm-1-928 cm-1, for guluronate fingerprint (G) at wave number 890.21 cm-1, for manuronate fingerprint (M) at wave number 847.88 cm-1, for methylene group (-CH2-) at wave number 732.46 cm-1, for thiol group (CH2-S(C-S-)) at wave 699.72 cm-1, for thioether group (CH2-S(C-S-)) at wave number 656.55 cm-1, for alkyne group (C-H) at wave 629.72 cm-1, and for aliphatic bromo group (C-Br) at wave number 602.79cm-1.

Based on the data in Table 1, the average setting time for the commercial alginate control group was 1 minute 39 seconds, while the average setting time

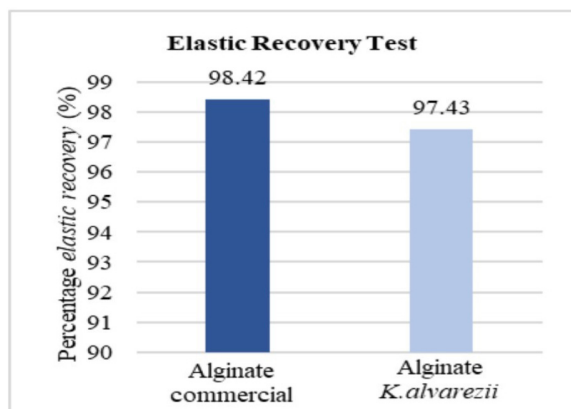


Figure 2. Histogram of the mean results of elastic recovery measurements.

for the treatment group, was 3 minutes 30 seconds. Furthermore, Table 2 shows the average elastic recovery value of 98.42% in the control group and 97.43% in the treatment group.

The elastic recovery test data results were analyzed using SPSS computer software as a normality test, homogeneity test, and T-test. After the normality and homogeneity tests were carried out, the data were found to be normally distributed and homogeneous. The T-test results obtained sig. (2-tailed) of 0.122 ($p > 0.05$), which showed a difference but was not significant in the results based on the test performed. Figure 2 is a histogram showing that the average elastic recovery of the treatment group (*K.alvarezii alginate*) of lower than that of the control group (commercial alginate).

DISCUSSION

Based on the elastic recovery test, there is no significant difference between the sample groups. This is because the alginate impression material derived from the red algae (*K. alvarezii*) has similarities to commercial alginate impression materials made from brown algae in the content and the primary source of gelling sodium alginate. Alginate consists of hydroxyl, carbonyl, carboxyl groups, manuronate, and guluronate fingerprints. In the FTIR analysis results of alginate from red algae, hydroxyl groups (O-H) were obtained at 3381.39 cm⁻¹, carbonyl groups (C=O) at wave number 1634.55 cm⁻¹, carboxylate ions (COO⁻) at wave number 1323.23 cm⁻¹, guluronate fingerprints (G) at wave number 890.21 cm⁻¹, and manuronate fingerprints (M) at wave number 847.88 cm⁻¹. The results of the functional group analysis in this study were almost the same in the study of Kamisyah et al., who performed FTIR analysis on alginate from brown algae with the (O-H) group at 3442.93 cm⁻¹, carbonyl group (C=O) at 1645.28 cm⁻¹, carboxylate ion (COO⁻) at 1319.31 cm⁻¹,

and manuronate fingerprints at 873.75 cm⁻¹.¹⁸ Based on the FTIR analysis results, it can be concluded that the compound contained is alginate, which can form a gel. In alginate impression materials, the gel forms a solid consistency and elastic mass to produce negative molds of teeth and surrounding tissues in the mouth.¹⁹

Their composition influences the setting time of alginate impression materials. The content of trisodium phosphate, as an inhibitor, determines the gel formation time, while calcium sulfate is the reactant that promotes gel formation. Mixing the alginate impression material with water causes a slow reaction phase (polymerization process) at the beginning of the reaction between sodium phosphate and calcium sulfate. The remaining calcium sulfate reacts with sodium alginate to form insoluble calcium alginate which forms a gel with water, which acts as a catalyst.² The composition of the retarder must be carefully adjusted to ensure that the formation time is adequate.²⁰ Therefore, the powder-to-water ratio is important to speed up or slow down the reaction.

The shelf life of impression materials in a package had an effect on the reduction or increase of water content by 15%. Reduced water content in the impression material for longer shelf-life results in a faster setting time and higher viscosity than standard water content. Therefore, the ratio of dental alginate powder to water depends on the condition of the impression material.²¹ This may explain why factory-made alginate impression materials have a faster setting time due to the shelf-life purposes of the packaging at the point of sales, which allows the water content of the content to be reduced.

The particle size of the alginate impression material affects polymerization. Based on previous studies, alginate impression materials, whose sodium alginate is derived from red algae, have higher porosity than manufactured alginate due to the larger particle size. In addition, the source of the alginate also influences the setting time. In this study, sodium alginate was extracted from red algae, while the alginate material from the manufacturer was likely derived from brown algae. Red algae contain less sodium alginate than brown algae. Fourier Transform Infrared (FTIR) analysis showed the same wave absorption pattern, although there was a slight shift. Some materials used during extraction were absorbed, causing a shift in the absorption peak, and affecting the quality of the alginate. Therefore, it is necessary to identify the quality of the results of sodium alginate extraction from red algae in more detail. These accompanying components have caused ionic reactions that affect the alginate reaction phase, thus potentially affecting the setting time. The quality and quantity of bioactive ingredients in algae, including polysaccharides, depend on location, origin, species, and harvest time.⁶

The elastic recovery of alginate from red algae *K. alvarezii* was not significantly lower than that of Hygedent alginate impression material. The elastic properties of the impression material influence the elastic recovery. The manuronate and guluronate monomer content of sodium alginate can influence the elasticity of the alginate impression material. Alginate with a higher polyguluronate content (a collection of guluronate monomer chains) will produce a stiff gel structure. In comparison, alginate with a higher polymanuronate content (a group of manuronate monomer chains) will form a more elastic gel.²² Commercial alginate impression materials made from brown algae, such as *Sargassum sp.*, contain sodium alginate with a higher ratio of manuronate monomers. Khajouei et al. explained that the ratio of manuronate monomer in sodium alginate from the algae *Saragassum sp* is greater than that of guluronate monomer.¹⁰ This makes the material structure less stiff and more elastic, so the elasticity of commercial alginate is slightly higher and insignificant.²³ The impression material sodium alginate from *K. alvarezii* tends to have a higher structural content of guluronate monomer than manuronate. Alginate produced from algae in regions with tropical climates (warm water), such as Indonesia, tends to have a higher guluronate content.²⁴ The higher the content of the guluronate block in the alginate, the higher is the ability of the alginate to bind Ca²⁺ ions.²⁵ When mixed with divalent cations such as calcium, alginate form strong interactions between calcium ions and COO- groups of guluronic acids of different chains, thus creating a three-dimensional lattice often referred to as an "egg box" structure.¹⁰ The greater the number of cross-linked complexes that are formed from Ca²⁺ ions and polyguluronic chains, the stronger the resulting gel will be. By manipulating the water absorption, the alginate impression material can change the sol structure into fibrils. The fibril structure will then intertwine to form a brush heap structure further and form a gel. This brush heap structure is due to the cross-linking of molecular complexes. The cross-linking reaction occurs between sodium alginate in the polyguluronic chain and calcium sulfate (CaSO₄) as a source of calcium ions (Ca²⁺). The content of calcium ions and polyguluronate chains makes the gel form more rigid (increasing gel rigidity) so that the elasticity of the material is reduced, which results in lower elastic recovery of the material.²⁶

The method of impression manipulation is one of the factors that can affect elastic recovery. A study conducted by Frey et al. showed that mechanical manipulation of alginate impression materials can improve the elastic recovery properties of the materials compared to manual alginate mixing techniques.⁷ Mechanical manipulation produces a good and smooth mixture and reduces air bubbles that can form voids, thereby increasing the elastic recovery of the materials. In this study, the ingredients use the hand mixing method (manual), affecting the amount of elastic

recovery of materials that become lower.²⁷ In a study by Maiola et al., mechanical mixing of impression materials on commercial alginate impression materials resulted in an elastic recovery of 99.0%. While the manual method in this study manipulated commercial alginate, an obtained a lower elastic recovery of 98.42% was obtained.¹⁹ Also, the alginate impression material made from red algae *K. alvarezii* had a lower elastic recovery than the control group, possibly due to the manual manipulation method.

During manipulation, the alginate impression material absorbs water, which can change the sol structure into fibrils. The fibril structure will intertwine with each other, continuing to form a brush pile structure and forming a gel. This brush pile structure is due to the complex cross-linking of the molecules. The cross-linking reaction occurs between sodium alginate in the polyguluronic chain and calcium sulfate (CaSO₄) as a source of calcium ions (Ca⁺). The content of calcium ions and polyguluronate chains makes the formed gel stiffer (increases gel stiffness), so that the elasticity of the material is reduced and results in lower elastic recovery of the material.²⁶ The method of impression material manipulation is a factor that can influence elastic recovery. A study by Frey et al. showed that mechanical manipulation of alginate impression materials can improve the elastic recovery properties of the material compared to manual mixing techniques.⁷ Mechanical manipulation produces a good and smooth mixture and reduces air bubbles that can form voids, which causes the elastic recovery of the material to be higher. In this study, the material manipulation method used was the manual hand mixing method, which resulted in the lower elastic recovery of the material.²⁷ In a study by Maiola et al., the mechanical method of mixing impression materials on commercial alginate impression materials resulted in an elastic recovery of 99.0%,¹⁰ while the manual method resulted in a lower elastic recovery of 98.42%.¹⁹ The low elastic recovery in this study may also be due to manual manipulation. Elastic recovery is also influenced by the setting time of the impression material, as elastic recovery increases significantly within two to three minutes after the material is properly manipulated and set.⁷ Research has shown that the setting time of commercial alginate impression materials is faster than that of *K. alvarezii* alginate impression materials. This may explain why the longer the setting time of the impression material, the lower the elastic recovery will be.²⁸

CONCLUSION

Based on the results of the study, it can be concluded that the alginate impression material based on red algae *K. alvarezii* has an elastic recovery of 97.43% . which is lower than that of the commercial alginate impression material.

CONFLICT OF INTEREST

The authors declare no conflict of interest with respect to this study.

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