ORIGINAL ARTICLE

The value of imbibition and syneresis for dental impression on red seaweed: a laboratory experiment

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ABSTRACT

Introduction: Alginate is a type of elastic dental impression, which is an irreversible hydrocolloid. The main components in sodium alginate can be obtained naturally through the extraction process from seaweed *(Kappaphycus alvarezii)*. Several physical characteristics must be fulfilled by dental impressions, such as dimensional stability related to imbibition and syneresis. ANSI/ADA specification No.18 regarding the maximum permitted change in alginate impression materials may not exceed 0.5%. This research aimed to analyze the value of imbibition and syneresis on dental impressions of red seaweed extract *(K.alvarezii)*. **Methods**: The research was conducted using an experimental laboratory method with a post-test-only control group design. There were two groups, namely the control group of commercial dental impressions and the treatment group using dental impressions with the sodium alginate extracted from red seaweed *(K.Alvarezii)* by the acid method, and the FTIR test was performed to analyze compound sodium alginate. Imbibition and syneresis measurements were carried out using a calliper on a plaster cast model from negative mould casting results, where previously, the mould was soaked in water for imbibition and evaporated for syneresis. **Results**: Imbibition and syneresis tests showed that the impression material group with sodium alginate from red algae was significantly lower than those with commercial impression materials. It was concluded that the impression material with sodium alginate from red seaweed had an effect on reducing the percentage of syneresis ($p \leq 0.05$) and imbibition ($p \leq 0.05$) of the impression material. Based on the results of our research, it was found that the alginate impression material extracted from red seaweed *(k.alvarezii)* had an imbibition value of 0.33% ($p \leq 0.05$) and syneresis of 0.28%, ($p \leq 0.05$) lower compared to alginate impression materials. commercial alginate impression material. **Conclusion**: The imbibition and syneresis values of red seaweed alginate show an excessively low value compared to commercial alginates.

KEYWORDS
dental impression,sodium alginate, *Kappaphycus alvarezii*, imbibition, syneresis

INTRODUCTION

In dentistry, impression materials are often used in making imprint of teeth and surrounding tissues. Impression materials are often divided into two categories: non-elastic materials (i.e, impression compound, impression wax, and zinc oxide eugenol) and elastic materials (i.e, elastomer, reversible and irreversible hydrocolloids).[1] Alginate is one of the most widely used material for impression because it is easy to use and economical, making Indonesia as one of the alginate importing countries.[2] The main component of alginate is sodium alginate which can be obtained naturally through an extraction process.[3] Sodium alginate is one of a group of natural polysaccharides that is found in the cell walls of various types of seaweed species.[4]

Based on their pigments, seaweeds are divided into three categories, namely *Rhodophyceae* (red seaweed), *Chlorophyceae* (green seaweed), and *Phaeophyceae* (brown seaweed). *Rhodophyceae* (red seaweed) has the potential to be developed as raw material for the alginate industry.[5] The amount of polysaccharide contained in red seaweed is the highest compared to the others.[6] Polysaccharides in red seaweed consist of alginate, agar, and keragenan.[7,8] Agar and carrageenan are used as gelling agents, binders, stabilizers, and syneresis inhibitors (preventing water release).[9] The alginate contained in polysaccharides can be extracted into sodium alginate, and become the raw material for dental impression materials.[10]

Alginate impression materials' physical and mechanical characteristics must comply with predetermined standards, including dental impressions, such as good elastic recovery, dimensional stability, and mechanical properties.[11] Red seaweed alginate studies revealed elastic recovery values that were comparable to those of commercial alginates.[12] This led the teams to discover more about other characteristics of alginate red seaweed, such as dimensional stability. Dimensional changes are characterized by imbibition and syneresis. Imbibition is a change in shape that occurs when the impression material is immersed in water for a specific time so that it expands, which can cause distortion. Syneresis occurs if the dental impression material experiences evaporation and shrinkage, making the results inaccurate.[13,14]

According to Nurliyani's research (2022) which refers to ADA specification no 18 related to the maximum change allowed for alginate impression materials which should not exceed 0.5% from the original size.[15,16] If the imbibition and syneresis of alginate impression materials exceed the maximum limit standards, the resulting study model has poor dimensional stability, allowing failure during dental procedures. Based on this explanation, the purpose of this research is to analyze the value of imbibition and sineresis of dental impressions of red seaweed extract *(K.Alvarezii).*

METHODS

This research used an experimental laboratory type with a post-test only control group design conducted in the Bioscience and TKG laboratories of RSGM Jember University.There were two groups used, namely the imbibition and syneresis control groups using commercial dental impressions and the imbibition and syneresis treatment group using dental impressions with sodium alginate extracted from *K.Alvarezii.*

Wet *K.Alvarezii* seaweed was washed with tap running water then soaked in 0.1% KOH for 1 hour. Samples were washed again and then dried at 60^0 in the oven until the moisture content was <15%. Pulverization was done using a blender until it became powder and weighed as much as 100 grams, soaked in 1% HCl solution (1:30 b/v) for about 1 hour and then washed until the pH became neutral. Extraction process was for 2 hours with 2% Na_2CO_3 solution (1:30 b/v) using an ultrasonic waterbath at 60-70°C. The filtrate was purified with 10% NaOCl with as much as 4% of the filtrate volume for 30 minutes until it looked ivory yellow in colour. Precipitation of alginic acid was done by adding 10% HCl to the filtrate until the pH became 2.8-3.2. The precipitate was converted to sodium alginate with Na_2CO_3 until the pH became neutral, then added into isopropyl alcohol solution (1:2 v/v) while stirred well and waited for 30 minutes. The resulting sodium alginate was dried using….? at 60^0 in the oven and pulverized.[17]

The extracted sodium alginate was tested using an Fourier Transform InfraRed (FTIR) spectrophotometer to show the absorption peak. Powdered sodium alginate samples were inserted into the plate and analysed. The absorption peaks of the FTIR results were analysed to determine the functional groups of sodium alginate (Figure 1).

For the control group (commercial alginate impression material), the ratio of water and powder according to the manufacturing instructions was 5 grams: 11.5 ml of water and for the test group (red algae-based alginate) the ratio was 5 grams: 11.5 ml of water, then mixed using the hand method. The mixture was poured into a ring tube (diameter 20 mm), then the master die (diameter 5 mm) was inserted into the alginate mold until a negative mold was obtained and the diameter was measured with a caliper. In this study the number of samples used was 16 which were divided into a control group and a test group

In order to observe the imbibition property, the negative molds of the control and treatment groups were immersed in water for 12 minutes, and then gypsum plaster of paris was poured into the negative mold to produce positive model, which were subsequently measured using a caliper.[18]

While the sineresis property was observed by the following method: the negative molds of control and treatment groups were placed outdoors for 30 minutes, then gypsum plaster of paris was poured into the negative mold to produce positive models, which were subsequently measured using a caliper. The formula of dimension change for dental impression. [18]

The change in dimensions of the printing material (A-B) in mm is obtained from reducing the size of the master die (A) with the resulting gypsum model print (B) multiplied by 100%.

RESULTS

This research produced alginate powder based on Kappaphycus alvarezii seaweed extract. The FTIR test of a sodium alginate (C6H7O6Na) sample is shown in the following figure 1:

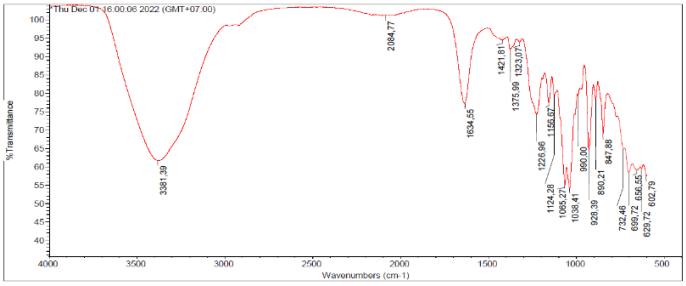

Figure 1. The result of FTIR spectrophotometry test

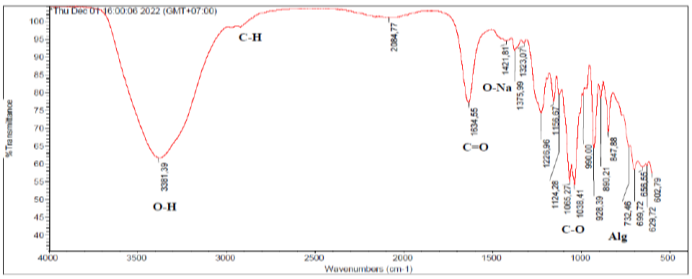

Figure 1. FTIR spectrophotometry of red seaweed sodium alginate

The figure shows the FTIR spectrum in the form of absorption peaks from the sodium alginate functional group. The specific absorption peak for the hydroxyl group (O-H) was around 3500-3200 cm-1; the alkyl group (C-H) was between 2840-3000 cm-1; the carbonyl group (C=O) was between 1600-1700 cm-1, and the carboxyl group (C-O) was between 1000-1300 cm-1 (Pasanda, 2020). Alginate also had a typical guluronic fingerprint (α-L guluronic acid (G)) in the absorption area of 890–900 cm-1, a typical mannuronic fingerprint (β-D mannuronic acid (M)) in the absorption area of 810–850 cm-1, as well as sodium bonds between 1614-1431 cm-1.[19]

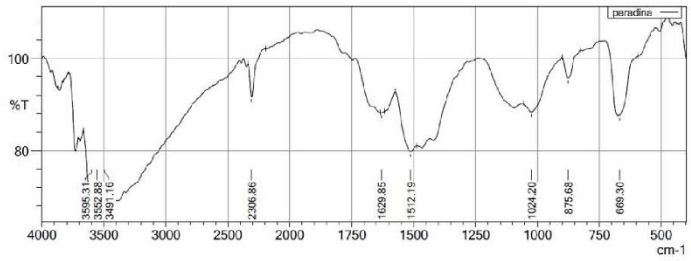

Figure 1. FTIR spectrophotometry of industrial alginate

The figure shows the FTIR spectrum of the functional groups for sodium alginate. The absorption peaks were specific for hydroxyl groups (O-H) at around 3500-3200 cm^{-1}; alkyl groups (C-H) were between 2840-3000 cm^{-1}; carbonyl groups (C=O) were between 1600-1700 cm^{-1}, and carboxyl groups (C-O) were between 1000-1300 cm^{-1}.[19] Alginate also had typical fingerprints of guluronate (α-L guluronic acid (G)) at 890-900 cm^{-1}, manuronate (β-D manuronic acid (M)) at 810-850 cm^{-1}, and sodium bond at 1614-1431 cm^{-1}.[20] Based on the results of the FTIR spectrum, the functional groups in the extracted powder of red seaweed *K.alvarezii* matched with the characteristics of sodium alginate.

Table 1. Average of imbibition test measurements

Sample	Control (%)	Treatment (%)
1	0.40	0.20
2	0.60	0.40
3	0.53	0.40
4	0.47	0.33
Average	0.50	0.33

Table 2. Average of syneresis test measurements

Sample	Control (%)	Treatment (%)
1	0.47	0.27
2	0.53	0.20
3	0.60	0.27
4	0.53	0.40
Average	0.53	0.28

The imbibition test results for the control group was 0.50%, while the treatment group had an average value of 0.33% (table 1). In the measurement results of the syneresis test, the average for the control group was 0.53%, while in the treatment group it was 0.28% (table 2). The data results were then analyzed with SPSS including normality test, homogeneity test, and t-test. The data were normally distributed and homogeneous. In the parametric t-test, significant differences were obtained in the control and treatment groups, with a sig value of 0.039 ($p \leq 0.05$) for the imbibition test and 0.002 ($p \leq 0.05$) for the syneresis test.

DISCUSSION

In the imbibition test, the tooth impression extracted from Kappaphycus alvarezii obtained a value of 0.33%, lower than the commercial alginate impression material of 0.50% which can be seen in table 1. The difference in imbibition values is influenced by several factors such as particle density, type and location of grass growth sea, polysaccharide content, and number of carboxylate ions. The imbibition process occurs when alginate binds with water and then the parallel polysaccharide chains separate. This progresses from low to high concentration without involving the semipermeable membrane until osmotic balance is achieved between the water and the tooth mold.[21]

Particles of dental impression that are dense cause the time needed to fill the cavity in the dental impression is longer so that the resulting imbibition is also lower.[22] A lower imbibition value tends to indicate a more dimensionally stable quality of the impression materials. The majority of commercial alginate impression materials are imported from countries with subtropical climates using sodium alginate from brown seaweed, while the alginate impression materials in the treatment group coming from red seaweed (*K. alvarezii)* were taken from the sea in Banyuwangi with a tropical climate. Sodium alginate from tropical seaweed (warm water) has an advantage in the gel formation process associated with high polysaccharide contained. *K. alvarezii* with high polysaccharides can produce longer polymer chains with tighter bonds so that they can slow down the entry of water into the dental impression.[18,23]

The ability of dental molds to bind water depends on the amount of carboxylate ions. Based on Figures 1, it can be seen that the absorption of the COOH group (carboxyl group) when released from the alginate polymer chain would cross-link with the water group.[24] In the carboxylate group there is an –OH component which is hydrophilic so it will react quickly with water which is able to form hydrogen bonds. According to Febriana (2018), increasing the number of carboxylate ions will increase the imbibition value of the alginate impression material.[25] Based on this explanation, it is suspected that brown seaweed had a greater number of carboxylate ions so that the imbibition value was greater.[26] compared to tooth casts extracted from K. alvarezii.

The spectrum of sodium alginate extracted from red seaweed (figures 1) had similar results to sodium alginate of Sargassum tenerrimum (Iqra Rasheed, 2018). The absorption peak is similar between 1500 cm to 1000 cm which indicates the presence of hydroxyl groups and the absorption peak is 1000-750 which indicates the presence of Alg groups.[26,31]

Syneresis test results for alginate impression material extracted from Kappaphycus alvarezii showed a value of 0.28% lower than commercial alginate impression material, which can be seen in table 2. Syneresis is related to the water release process which is characterized by shrinkage of the gel from the tooth impression due to several factors such as the calcium ratio , manipulation technique, W/P ratio, and molecular weight. On the other hand, if the alginate releases a lot of Ca2+ ions during gel formation, more cross-links can form between molecules. The more cross-links, the syneresis process that occurs will also increase.[27 30]

Brown seaweed contains higher calcium (10.3 g/kg) than red seaweed (3.11 g/kg).[26] The Ca2+ concentration during the alginate gel formation process can influence the characteristics of the tooth cast.[27] With the low calcium ratio in the tooth cast, alginate will lose less water, resulting in lower syneresis values. Imbibition and syneresis also depend on composition, molecular weight, calcium concentration, storage and environmental conditions.[28]

The syneresis occurrence in the control group of commercial alginate impression materials had a higher value and exceeded the maximum limit with a difference of 0.03%. Based on ADA standard no. 18 regarding the maximum recommended maximum limit of dimensional change, which is 0.5%. The syneresis test in the control group that exceeded the limit could be affected by the manipulation technique. The researcher used the hand-mixing method because the extraction results were limited so that they could not be mixed evenly when manipulated using a vacuum mixer. The choice of method during research can affect the dimensional stability of the alginate impression. According to Zahid.[29] Alginate impression materials produced by automatic manipulation methods have better dimensional accuracy than using hand-mixing method. The mixing technique with a vacuum mixer produces a homogeneous mixture with almost no porosity. Hand-mixing method requires proper mixing time and figure-8 motion which is the best way to stir the mixture so that it minimizes the size change in dental impression.[30]

In our preliminary research (Indahyani 2023), the W/P ratio affected dimensional stability (imbibition and syneresis), due to differences in viscosity between the control and treatment groups even though the W/P ratio was the same. Viscosity affects the molecular mass of the alginate polymer. Molecular mass is directly proportional to viscosity; if the molecular weight increases, the viscosity increases. This is in accordance with research by Pratiwi, which explains that the viscosity value of commercial alginate is higher compared to alginate extracted *from K.alvarezii*.[15] The increase in viscosity is due to the large attractive force between the molecules bonding to each other. In syneresis, the molecular bonds become stronger due to air pressure between the polysaccharide chains, resulting in more water drops coming out (evaporation) from the dental impression.[26,27]

The limitations of the research that we found were that the extraction results were small, and in our study we also found an increased viscosity of the alginate obtained. These two factors require further research to get maximum results

CONCLUSION

The imbibition and syneresis values of red seaweed alginate show an excessively low value compared to commercial alginates.

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