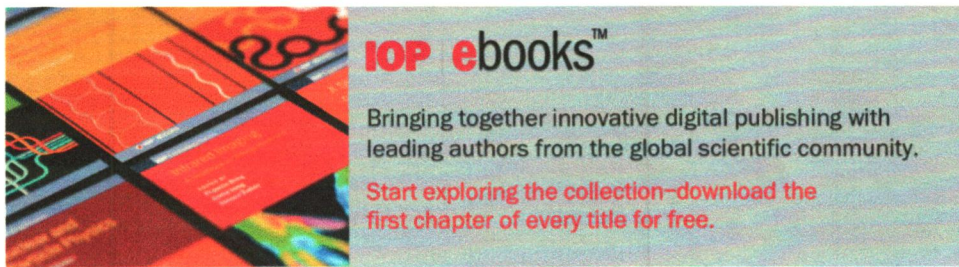


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Physical properties of irreversible hydrocolloid dental impression materials obtained from brown algae species *Padina* sp.

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use, the advantages of using alginate include an accurate reproduction of details, patient comfort, and the ease of cleaning and fabricating casts [1,2,3].

Sodium alginate, the main component of irreversible hydrocolloid impression materials, is a polymer composed of two monomer units of mannuronic and guluronic acids. The addition of water to sodium alginate results in the formation of a sodium alginate sol; calcium sulfate can be added to this solution to act as a reactant. Furthermore, the addition of diatomaceous earth and silica gel as filler materials increases the strength, hardness, setting time, and other physical properties of this impression material. Potassium sulfate and sodium or trisodium phosphates can accelerate or retard the setting time, respectively, whereas polyethylene glycol (PEG) maintains the stability of the impression material [3,4,5,6].

The use of the appropriate amount of the impression material produces accurate reprints of the oral cavity. However, it is imperative to take the following factors into consideration while preparing an impression material: setting time, working time, viscosity, compatibility with gypsum, dimensional stability, and filler compatibility [7]. In clinical situations, the hardening time taken by alginate tends to be too less (3–4 min); therefore, dentists have to modify the water-to-powder ratio of the impression material. The setting time of alginate can be manipulated by adding calcium phosphate or calcium sulfate, or by changing the water temperature [2,5,8].

Sodium alginate can be extracted from brown algae, especially *Padina* sp., which is one of the most abundant and economical brown algae found in Indonesian waters. These algae contain high amounts of chlorophyll, beta carotene, violaxanthin, fukosanthin, pirenoid, and filakoid [9].

Padina contains high levels of alginate that are sufficient to produce sodium alginate, the main component of dental impression materials (irreversible hydrocolloids) [4]. Several studies have been conducted, but the results have not been appropriately used for producing an effective dental impression material. The aim of this study was to produce a dental impression using irreversible hydrocolloid material obtained from the brown algae *Padina* sp. Several physical characteristics of alginate, such as purity, setting time, and viscosity, were tested and compared with those of different formulations of standard sodium alginate.

2. Methods

The materials used in this study included *Padina* sp., aquades, water, 5% HCl, 4% Na₂CO₃, 12% NaOCl, 10% NaOH, isopropanol (IPA), calcium sulfate, silica gel, potassium sulfate, PEG, diatomaceous earth, and trisodium phosphate.

2.1 Extraction of sodium alginate *Padina* sp.

The brown algae *Padina* was obtained from the Makassar Strait was used for extracting sodium alginate. Dried *Padina* sp. was soaked in 1% HCl solution for 1 h and washed with aquades three times. Na₂CO₃ (4%) was added and the mixture was heated at 60°C for 2 h while stirring until a paste-like consistency was reached. The mixture was then solubilized in aquades for approximately 30 min and filtered. The residue was bleached by adding 12% NaOCl solution while stirring until evenly distributed. Subsequently, 5% HCl was added (pH 2–3) to the filtrate, resulting in the formation of alginic acid. The mixture was then filtered to obtain foam clumps of alginic acid, which were washed with water to prevent the formation of harmful acid residues and 10% of this mixture was added to pH 10 NaOH. Next, 95% isopropanol was added to the alginic acid that was converted to sodium alginate, and this was frozen for 12 h. The separated sodium alginate was filtered and dried to extract the powdered form of sodium alginate *Padina* sp., which is used to prepare the sodium alginate impression material [3].

2.2 Preparation of the dental impression material (irreversible hydrocolloid)

The dental impression material was prepared by mixing all the ingredients (sodium alginate extracted from *Padina* sp., calcium sulfate, potassium sulfate, diatomaceous earth, silica gel, PEG, and

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Physical properties of irreversible hydrocolloid dental impression materials obtained from brown algae species *Padina* sp.

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Abstract. The aim of this study was to prepare an irreversible hydrocolloid dental impression material extracted from brown algae (*Padina* sp.). The physical properties, such as purity, viscosity, and setting time of the material, were evaluated and compared with those of standard alginate. This was a quasi-experimental study with a one-shot case study design using brown algae *Padina* sp. Sodium alginate was extracted from the brown algae *Padina* sp. and irreversible hydrocolloid dental impression material was prepared by mixing it with four different formulations of calcium sulfate, potassium sulfate, diatomaceous earth, silica gel, poly ethylene glycol, and trisodium phosphate. Tests for measuring the absorption (Fourier-transform infrared spectroscopy [FTIR]), setting time, and viscosity were performed to compare the physical properties between the *Padina* sp. and standard sodium alginates and impression materials. The FTIR spectrum of the *Padina* sp. alginate was similar to that of the standard dental impression materials, except for the absorption rate. The fastest setting time was 3.15 min, and the longest was 6.51 min. The mean viscosity of the *Padina* sp. dental impression materials ranged from 40 to 160 cP and that of the standard impression materials was 920–2400 cP. The FTIR spectra and setting times of the *Padina* sp. alginates were similar to those of the standard dental impression materials. Despite the low viscosity and flowability, the *Padina* sp. impression materials may be used for taking dental impressions.

1. Introduction

Alginate, which is the most widely used irreversible hydrocolloid in dentistry, is an elastic impression material that is generally used for prosthodontic and orthodontic purposes. In addition to the ease of



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 trinitrium phosphate) using a mortar and pestle. The impression material sample was prepared using four variations of the formulation (Table 1). A total of 20 formulations on the basis of the basic formulation by Putri WA (2013) were prepared [10,11]. The sample tests conducted included Fourier-transform infrared spectroscopy (FTIR), setting time, and viscosity.

Table 1. The composition and formulation of alginate dental impression materials.

Standard alginate	Padina alginate (%)	Calcium sulfate (%)	Potassium sulfate (%)	Diatomaceous earth (%)	Silica gel (%)	PEG (%)	Trinitrium phosphate (%)	Total (%)
P1/S1	20	40	15	4	13	7	1	100
P2/S2	19	40	15	4	14	7	1	100
P3/S3	19	40	16	4	13	7	1	100
P4/S4	19	41	15	4	13	7	1	100

P, Padina.

S, Standard.

PEG, Polyethylene glycol.

2.3 FTIR

FTIR was conducted for analyzing the chemical composition of the organic compounds. Infrared spectra are used for determining the existence of several chemical bonds and organic compounds. Infrared spectroscopy techniques are primarily used for identifying the functional groups of a compound, determining its molecular structure, identifying the compound, evaluating its purity, and studying the ongoing reactions. FTIR data is displayed in the form of a transverse wave spectrum. Each peak or valley of the wave spectrum displays a number that represents the length of each functional group isomer of the wave spectrum. The absorption areas or wavelengths for each functional group of the sodium alginate compound were evaluated and comparisons were made between the standard and extracted sodium alginates. The quality of the sodium alginate obtained by the extraction of brown algae is equal to that of standard sodium alginate [12, 13].

2.4 Setting time

Four grams of each dental impression formulation were mixed with 9.5 ml of distilled water for 1 min at 24°C. The mixture was placed on the impression and gel formation time was recorded using a stopwatch. The setting time was recorded when the impression material lost its stickiness; it is crucial to accurately record this time because it will help the dentist in determining the amount of time required to mix the material, fill the impression tray, and place it in the patient's mouth [4].

2.5 Viscosity

Viscosities of the samples were measured using the LV Brookfield viscometer. The sample of a finely powdered printing material was weighed using a digital scale and then mixed with water at a ratio of 1:1. The water (100 ml) was heated to varying degrees (75°C, 70°C, and 65°C) following which the material was added and stirred with a homogenizer. The ingredients were individually tested on the test machine by placing the beaker containing the sample solution in the viscometer machine. Spindle 5 was used for thick materials and spindle 3 for dilute materials. The test was performed three times for each sample.

During this data-processing stage, data matching was performed by converting the values according to the following formula: results obtained from the viscometer multiplied by the multiplier factor on the basis of the speed and spindle used.

3. Results

The FTIR spectra of the sodium alginate and impression materials (*Padina* sp. vs. standard) are shown in Figures 1 and 2.

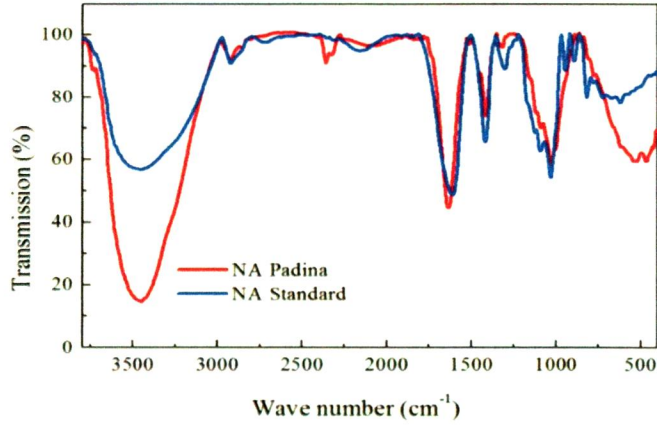


Figure 1. Infrared spectrum of sodium alginate *Padina* sp. and standard sodium alginate.

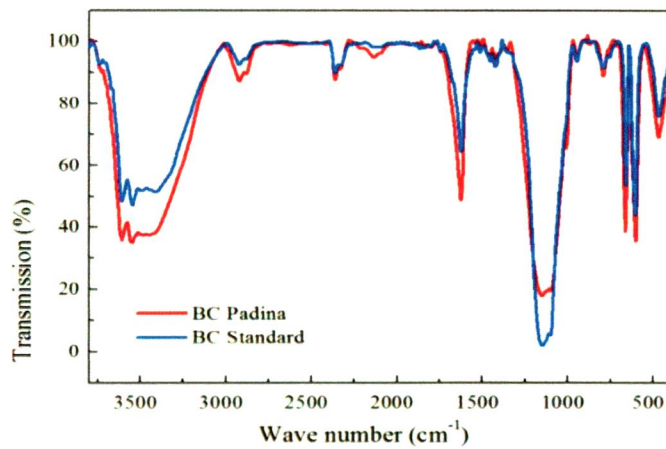


Figure 2. Infrared spectrum of alginate impression material *Padina* sp. and standard impression materials.

The setting times of the *Padina* sp. and standard impression materials are presented in Figure 3.

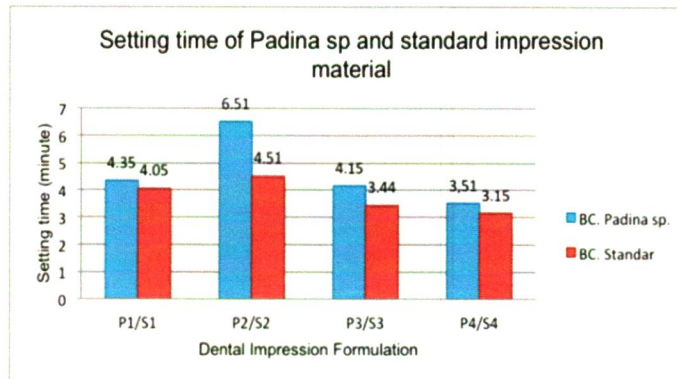


Figure 3. Graph showing mean setting times of the *Padina* sp. and standard impression materials. P1–P4, *Padina* sp. impression material formulae 1–4. S1–S4, Standard impression material formulae 1–4.

As observed in the graph, the mean setting time of *Padina* sp. impression material P4 was the fastest at 3.51 min, followed by those with P3 and P1. The longest setting time was noted with P2 (6.51 min). The setting times of *Padina* sp. impression material formula P1 were similar to those of the standard impression material formula S1 at 4.35 and 4.05 min, respectively.

A comparison of the mean viscosities of the *Padina* sp. and standard alginate impression materials is shown in Table 2.

Table 2. Mean viscosity of irreversible hydrocolloid impression material from brown algae-based *Padina* sp.

No	Dental Impression	Viscosity (cP)	Spindle (No)	Rate (rpm)
1	P1	160	3	50
2	P2	40		
3	P3	40		
4	P4	40		
5	S1	2400	5	
6	S2	1280		
7	S3	920		
8	S4	960		

P1–P4, *Padina* sp. impression material (formulae 1–4).

S1–S4, standard impression material (formulae 1–4).

The results show that the rheology or flow type of the *Padina* alginate impression material is lower than those of the standard impression materials. This viscosity test uses the viscometer at a speed of 50 rpm. The *Padina* alginate impression materials were tested using spindle 3 because of the fluid consistency of the samples. The materials were not completely mixed when they were added to water, resulting in the deposition of some of the unbound material at the bottom of the beaker.

4. Discussion

In the present study, the physical characteristics of *Padina* sp. sodium alginate (purity, setting time, and viscosity) were tested and compared with those of different formulations of standard sodium alginate. The FTIR spectra of standard sodium alginate and impression materials were similar to those of the *Padina* sp. sodium alginate and impression materials. However, differences were noted in the degree of absorption. For example, the -OH bond at wave number 3500 cm^{-1} demonstrated a similar hydrogen bond with the hydroxyl group (-OH). Standard sodium alginate demonstrated a transmission of approximately 56%, whereas in the *Padina* sp. alginate it was approximately 14%, indicating that *Padina* sp. has the ability to bind to other molecules, such as water, much faster than standard alginate. In the second peak, the nitrite groups of the two samples were almost identical, and therefore, they did not affect the gelation process. At the C-C peak of aromatics, the aromatic carbon bonds appear to be present with different intensities of transmission.

In the FTIR spectrum, the value of 3467 cm^{-1} refers to the -OH group, thus indicating that there was some residual liquid present in the *Padina* sp. and that *Padina* sp. sodium alginate was slower to gel when compared with the standard sodium alginate (Figure. 1). The FTIR spectrum of the *Padina* sp. and standard alginate printing materials demonstrated similar -OH bonds in both alginate print materials. The C≡N or nitrite bonds appeared to be present only in the *Padina* sp. sodium alginate, suggesting the requirement for further purification, whereas after printing, the C≡N bonds were noted in all samples, including the standard alginate impression materials.

C-C bonds at 1420 cm^{-1} and 1620 cm^{-1} (aromatic numbers) indicated that the original odor of *Padina* sp. sodium alginate was higher than that of standard sodium alginate. After the formation of the impression materials, the bonds in the *Padina* sp. sodium alginate were approximately close to that of standard sodium alginate. The C-N bonds of the 1126 cm^{-1} aliphatic amines indicated that the standard sodium alginate were brighter than the *Padina* sp. samples; however, the brightness of the two impression materials were similar. The obtained sodium alginates were brownish yellow or greenish yellow in color and smelled fishy. According to Mushollaeni 2011, the color of the extracted brown algae sodium alginate ranged from yellow to light brown. The dark color of the alginate was caused by the presence of fucoxanthin. Thus, if the brown algae used was dark in color, the extracted sodium alginate would also be dark colored [13,14].

Working and setting times can be accelerated using warm water; nonetheless, it is preferable to use products that suit individual habits and use water at room temperature between 18°C and 24°C [9]. The optimum setting time is between 3 and 4 min at room temperature (20°C). The temperature of the water added to the alginate powder was $21^{\circ}\text{C} \pm 1^{\circ}\text{C}$. Setting time plays an important role in enhancing the performance of the dentist and contributing to patient comfort, so that the patient does not have to keep his mouth open for a prolonged period [10].

Factors such as the condition of the water (pH and salinity, light, depth of algae, and the presence of nutrients) can affect the viscosity of the sodium alginate; additional factors such as the morphology of the algae (thallus form) also affect the viscosity. A long thallus results in higher viscosity [15,16]. The molecular weight of sodium alginate also affects the viscosity of the impression material. The greater the molecular weight, the higher the viscosity [17]. Furthermore, the viscosity of *Padina* sp. sodium alginate can be increased by immersion in 0.1% potassium hydroxide (KOH) solution for 60 min [7].

5. Conclusion

On the basis of the results obtained in the present study, the physical characteristics of the *Padina* sp. alginate impression material resembled those of standard alginate impression materials. The viscosity or flowability of *Padina* sp. alginate was low; yet, it may be useful for the production of dental impression materials. However, additional tests related to strength, surface morphology, material decomposition temperature, rheology, and dimensional changes in setting and elasticity must be conducted to meet the standardization criteria of dental impression materials.

6. Acknowledgements

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