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by Nurlindah Hamrun

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To cite this article: Nurlindah Hamrun *et al* 2019 *IOP Conf. Ser.: Mater. Sci. Eng.* **515** 012025

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Studies on Surface Morphology of Irreversible Hydrocolloid Impression Material Based on Brown Algae Type *Padina* sp.

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Abstract. This study aimed to investigate the potential of brown algae (*Padina* sp.). The investigation focused on surface morphology of the brown algae as preparation for making sodium alginate-based impression materials to obtain standardized impression materials. The sample was a natural sodium alginate type *Padina* sp. taken from South Sulawesi, Indonesia. The extraction process was conducted to attain the sodium alginate extract. Subsequently, an irreversible hydrocolloid impression material was fabricated by mixing some of the impression material composition and sodium alginate from *Padina* sp. The test of physical characteristic was conducted by describing the surface morphology of impression materials using scanning electron microscope (SEM). The data analysis of the SEM images showed that there was a difference in the surface morphology structure of the impression materials of the brown algae of the type of *Padina* sp. and standard impression materials showing a rough surface. Furthermore, the particles had no binding with other particles for all samples.

Keywords: Impression material, alginate, surface morphology, *Padina* sp.

1. Introduction

Irreversible hydrocolloid impression material becomes an important material to produce a dental impression, as well as hard and soft tissues impression. Importantly, impression stage becomes the initial stage of all dentures manufacture process, including crown, bridge, and orthodontic appliances. Therefore, developing the fabrication process of the impression material especially alginate that is commonly used in dentistry is necessary to be conducted. Previous works reported that alginate has accuracy in denture manufacture, patient's comforts, as well as easy mixing and modification with simple equipment [1,2].

Physically, alginate is an irreversible elastic hydrocolloid dental impression material, that after mixed with a substance and a chemical reaction occurs, the alginate cannot return to its initial form. If sodium alginate is mixed with water, a solution is formed, and calcium sulfate can be added as an amplifier. Practically, diatom earth and silica gel are added as fillers to enhance the hardness, setting time, strength,



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and physical properties of alginate gel. Accelerator and retarder, potassium sulfate, and sodium or sodium phosphate, respectively, are needed to adjust the setting time of impression material. In the previous work, it is stated that impression material stability is maintained and kept from being volatile by adding polyethylene glycol [3,4]. Furthermore, alginate impression material is served as a negative cast from a dental and oral tissue. The preliminary cast is then cast with gypsum and a stone-like product, to obtain a study model, a dental, and oral tissue replica [4,5].

Regarding the preparation of alginate impression material, it is very important to produce it by employing Indonesian natural resources to reduce the production cost. Previous work reported that one of the most abundant marine resources in Indonesia is algae which are around 8.6% [4]. There are about 28 species of brown algae from six genera in Indonesian waters, including *Dyctyota*, *Padina*, *Hormophysa*, *sargassum*, *Turbinaria*, and *Hydroclathrus*. Two of those types are *Sargassum* sp. and *Padina* sp., which are the types of seaweed that are very abundant in Indonesian water and have economic value. Therefore, *Padina* sp. has the potential to be developed and utilized as a producer of sodium alginate, a raw material of alginate impression material [6].

There are several prerequisites in dental impression manufacture that have to be fulfilled including the material which must be dilute enough to adjust in an oral cavity and thick enough to remain on the impression tray during impression process in the oral cavity, the material changes into rubber-like form during a certain time period in the oral cavity, the hardened impression mold does not tear when removed from the oral cavity, and the dimensions must remain stable [7,8]. Therefore, it is very important to investigate the potential of *Padina* sp. by investigating the physical behaviors of surface morphology to obtain impression material that met the impression material standard used in dentistry clinical application. Furthermore, the elemental composition of the impression material is also essential to be explored.

2. Methods

2.1. Materials

In this study, the materials used were *Padina* sp., 12% NaOCl, distilled water, water, 4% Na₂CO₃, 5% HCl, isopropanol (IPA), silica gel, 10% NaOH, calcium sulfate, polyethylene glycol (PEG), potassium sulfate, trisodium phosphate, and diatom soil.

2.2. Extraction of sodium alginate *Padina* sp.

In this work, the brown algae *Padina* sp. achieved from Makassar Strait, Indonesia was extracted into sodium alginate. The dried *Padina* sp. was then soaked in HCl 1% in a beaker glass for 1 hour, then washed with distilled water three times and then added with Na₂CO₃ 4%. Then, it was heated at 60 °C for 2 hours until it became a paste. This mixture was then liquefied with distilled water for 30 minutes and then filtered. The filtrate was then bleached by adding NaOCl 12% solution while stirring evenly, then added with HCl 5% till it reached pH 2-3, and filtered to obtain alginic acid in the form of foam lumps. Foam lumps were washed with water to avoid harmful acidic residues and added with NaOH 10% until it reached pH 9. The alginic acid that had been converted to sodium alginate was then added with 95% isopropanol then frozen for 12 hours. Separated sodium alginate was filtered and dried. The extraction result was *Padina* sp. sodium alginate powder that would be processed into sodium alginate impression material.

2.3. Manufacture of reversible hydrocolloid dental impression material from brown algae *Padina* sp.

The manufacture of impression material was done by mixing all of the ingredients using mortar and pestle. Some samples of dental impression material were made of sodium alginate, whether from *Padina* sp. or standard alginate, calcium sulfate, potassium sulfate, diatom earth, silica gel, polyethylene glycol (PEG), and trisodium phosphate in the different formulation as shown in Table 1. In general, such formulation refers to the basic formulation that has been conducted by the previous works [9,10]. The composition formula of impression material was carried out by mixing all of the ingredients with mortar and pestle. The material composition consisted of sodium alginate extracted from *Padina* sp., calcium

sulfate, potassium sulfate, diatom earth, silica gel, polyethylene glycol, and trisodium phosphate. The sample of impression material was made with four variations of formula as shown in Table 1. The same composition and formulation were also made of standard sodium alginate [10].

Table 1. The composition formula of irreversible hydrocolloid dental impression manufacture from brown algae *Padina sp.* and standards

Padina Formulati on / Standard	Sodium Alginate <i>Padina</i> (%)	Calcium sulfate (%)	Potassium sulfate (%)	Diatom earth (%)	Silica gel (%)	PEG (%)	Trisodium phosphate (%)	Total (%)
I. P1/S1	20	40	15	4	13	7	1	100
II. P2/S2	19	40	15	4	14	7	1	100
III. P3/S3	19	40	16	4	13	7	1	100

P: *Padina* algae

S: Standard algae

2.4. Surface morphological characteristics of irreversible hydrocolloid dental material made of brown algae *Padina sp.* and standard

The surface morphological characterization of irreversible hydrocolloid dental material made of brown algae *Padina sp.* and standard was done by SEM-EDX (Scanning electron microscopy-energy dispersive X-ray) at the Central Laboratory, Universitas Negeri Malang. The SEM-EDX characterizations were undertaken to determine how to define the micro material structure, particle size, distribution, and elemental composition of the samples.

3. Results and Discussion

Figure 1 and Figure 2 show the respective SEM images and elemental mapping of irreversible hydrocolloid impression material based on Brown Algae type *Padina sp.* for formula 1 (P1), formula 2 (P2), and formula 3 (P3). Figure 3 and Figure 4 show the respective of irreversible hydrocolloid impression material standard for formula 1 (S1), formula 2 (S2), and formula 3 (S3). Meanwhile, Table 2 presents the elemental percent weight of irreversible hydrocolloid impression material based on Brown Algae type *Padina sp.* for formula 1 (P1), formula 2 (P2), and formula 3 (P3). Furthermore, Table 3 presents the elemental percent weight of irreversible hydrocolloid impression material based on Brown Algae type *Padina sp.* for formula 1 (P1), formula 2 (P2), and formula 3 (P3).

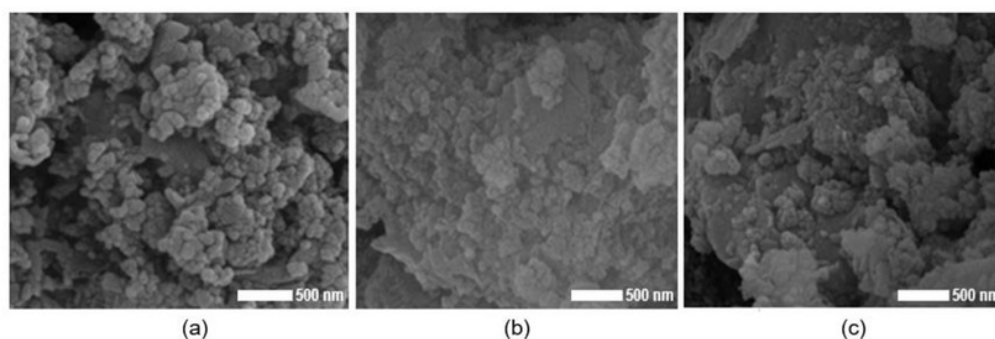


Figure 1. Surface morphology of irreversible hydrocolloid impression material based on Brown Algae type *Padina sp.* for formula 1 (P1), formula 2 (P2), and formula 3 (P3)

Based on the data, as can be seen clearly, the existence of Al, Fe, *etc* indicated the homogeneous samples, as we expected. SEM image was consistent with EDX results for all types of samples in this work. The homogeneous samples indicated the easiness to become slurry form and impression dental material applications. The XRD data (not shown) also exhibit the sharp peak which indicates the consistency of all chemical compounds in the samples. The sharp peak also indicated nano-micro particle existing for all composition in the samples. This data analysis showed consistent results among the SEM, EDX, and XRD data. Therefore, SEM, EDX, and XRD data exhibited that our samples have similar characteristics with standard samples indicating the same characteristics of dental impressions. The alginate impression material is a powder material. To use this material, it is mixed with water having various comparisons in accordance with the instruction of each producer [2]. The alginate impression material has the main components in the form of algin known as alginate acid or alginate.

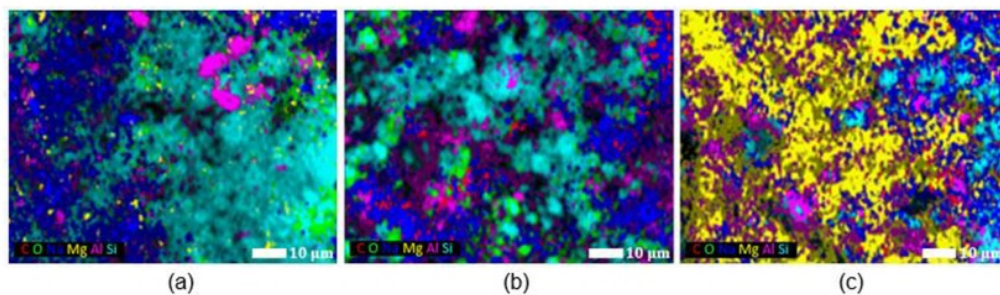


Figure 2. Elemental mapping of irreversible hydrocolloid impression material based on Brown Algae type *Padina sp.* for formula 1 (P1), formula 2 (P2), and formula 3 (P3)

Table 2. Elemental percent weight of irreversible hydrocolloid impression material based on Brown Algae type *Padina sp.* for formula 1 (P1), formula 2 (P2), and formula 3 (P3)

Wt%	C	O	Na	Mg	Al	Si	Cl	K	Ca	Fe	Cu	N
P1	5.15	37.97	5.65	1.78	4.96	26.79	3.86	1.83	8.94	3.08	-	-
P2	15.68	39.50	16.43	1.41	2.38	13.70	1.54	2.45	1.82	1.39	1.83	-
P3	13.52	36.07	12.76	-	5.51	10.67	5.07	1.71	6.73	2.88	-	5.09

Based on Figure 1 and Figure 3, almost all of both impression materials have a rough surface, and the resulted impression material was still weak in binding the other particles. This case was caused by some factors such as the quality of the samples used. Morphologically, the tallus form of each brown algae is different. This tallus form possibly influences the content of the resulted sodium alginate. The extraction method also gave effect on the quality of extraction result since the extraction process needs certain treatments such as the use of a reactor with a certain concentration, the number of the used reactor, pH of the solution, the temperature of heating and drying processes, the type of solution, and precipitator. Besides, the component of the impression material, the size of the less smooth particles, and the less homogenous mixing technique also influenced the results in making the impression material of irreversible hydrocolloid. According to Figure 2 and Figure 4; Table 2 and Table 3, it can be seen that the elemental composition constructed the samples are dominated by C, O, Na, Mg, Al, Ca, Si. Meanwhile, other elemental contents are detected as minority elements.

Associated with such data, the SEM characterization of phosphate zinc dental cement without the addition of ZnO nanoparticles showed the cracks. The cracks then reduced in the phosphate zinc cement that had been added with 0.1 gram of ZnO nanoparticles. Another significant factor was the stage of mixing the phosphate zinc dental cement with the heterogeneous ZnO also influenced the characterization result. That analysis relied on the ZnO nanoparticles having the nanometer size that fill the empty space between the big size-phosphate zinc particles. In the cooling step, the dental cement of the big size-phosphate zinc particles contacted with not only the big particles but also the small ones. This case caused the reduction of cracks and the increase in mechanical characteristic [11].

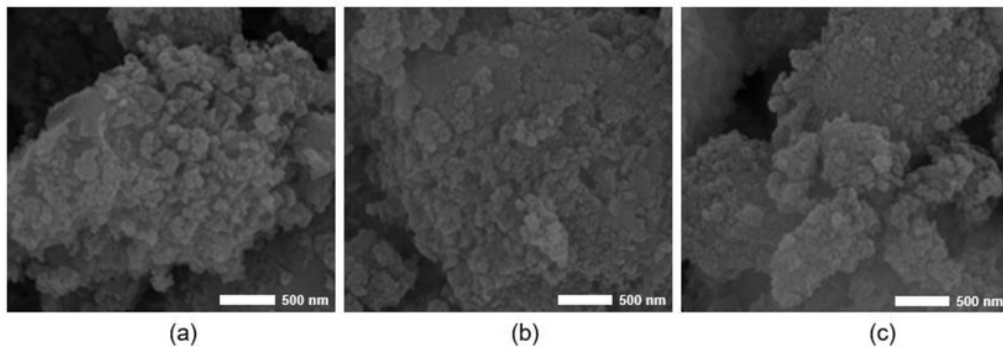


Figure 3. Surface morphology of irreversible hydrocolloid impression material standard for formula 1 (S1), formula 2 (S2), and formula 3 (S3)

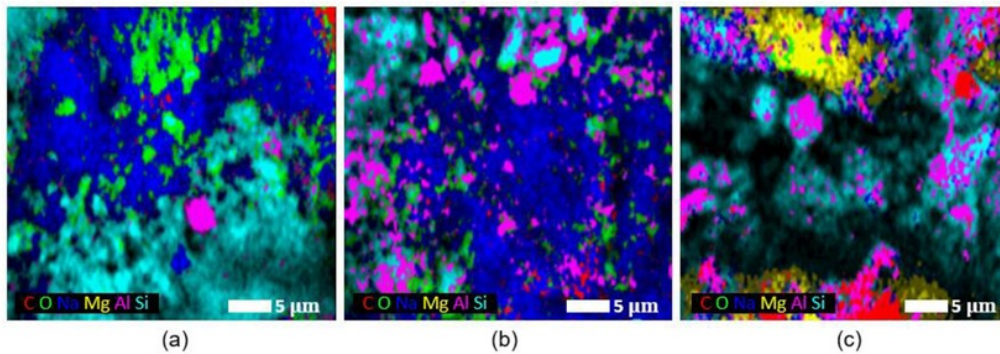


Figure 4. Elemental mapping of irreversible hydrocolloid impression material standard for formula 1 (S1), formula 2 (S2), and formula 3 (S3)

Table 3. Elemental percent weight of irreversible hydrocolloid impression material based on Brown Algae type *Padina* sp. for formula 1 (P1), formula 2 (P2), and formula 3 (P3)

Wt%	C	O	Na	Mg	Al	Si	Cl	K	Ca	Fe	Cu	Zn
P1	13.75	35.87	13.70	1.51	2.56	17.32	2.22	1.56	2.32	1.56	2.09	1.30
P2	15.68	39.50	16.43	1.41	2.38	13.70	1.54	2.45	1.82	1.39	1.83	-
P3	6.55	42.13	6.85	-	3.20	26.94	2.43	1.63	3.14	2.35	2.87	-

Another researcher tried to modify by adding cassava starch to the alginate. As shown in the previous work, the addition of cassava starch until 47.5% to the packaged alginate powder still resulted in detailed reproduction [5]. Moreover, the research conducted by another group also showed that the dimension stability of alginate impression material added with cassava starch with the ratio of 1:1 had the value of dimension stability fulfilling the standard of ANSI/ADA no. 18 that is the impression material may not change the dimension more than 0.5% of the model master. Furthermore, the addition of cassava starch with the concentration of 75% fulfilled the standard of ANSI/AD in term of stiffening period and the addition of cassava flour with the concentration of 50% met the standard of recovery from deformation of ANSI/ADA [12-13].

4. Conclusion

Based on the results and discussion, it can be concluded that the data analysis of the SEM images show a difference in the surface morphology structure of the impression materials of the brown algae with the type of *Padina* sp. and standard impression materials showing a rough surface. Furthermore, the particles had no binding with other particles for all samples. The SEM, EDX, and XRD data signified that the samples in this work have similar characteristics with standard samples indicated same characteristic dental impressions. Next, the extraction method also gives effects on the quality of extraction results. Another significant factor is the stage of mixing the phosphate zinc dental cement with the heterogeneous ZnO also influenced the characterization result. Moreover, the existence of Al, Fe, etc. indicated the homogeneous samples and SEM image was consistent with EDX results for all types of samples.

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Acknowledgements

The author would like to thank the Ministry of Research, Technology, and Higher Education through College Grant Award number 005/ADD/SP2H/LT/DPRM/VIII/2017 for financing this research.

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