

Research Report

Changes in setting time of alginate impression material with different water temperature

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ABSTRACT

Background: Previous studies showed that setting process of alginates can be influenced by temperature. **Purpose:** To determine the changes in setting time due to differences in water temperature and to determine the correlation between water temperature and the setting time. **Methods:** Seven groups of dough alginate were prepared by mixing alginate powder and water, each using a temperature between 13° C–28° C with a interval of 2.5° C. A sample mold ($\theta = 30$ mm, $t = 16$ mm) was placed on a flat plate and filled with dough alginate. Immediately the flat end of a polished acrylic rod was placed in contact with the surface of dough alginate. Setting time of alginate was measured from the starting of the mix to the time when the alginate does not adhere to the end of the rod. Setting time of alginate data were analyzed using one way ANOVA, LSD and Pearson. **Results:** Setting time of alginate with water temperature between 13° C–28° C were 87 to 119.4 seconds and were significantly different ($p < 0.01$). The setting time between group were also significantly different ($p < 0.01$). There was an inverse correlation between water temperature and the setting time ($r = -0.968$). **Conclusion:** Water temperature between 13° C–28° C with a difference of 2.5° C produced significant differences in alginate setting time; the lower the water temperature being used the longer the setting time was produced

Key words: Alginate, water temperature, setting time, polimerization

ABSTRAK

Latar belakang: Penelitian-penelitian sebelumnya menunjukkan bahwa proses pengerasan alginat dapat dipengaruhi oleh suhu. **Tujuan:** Mengetahui perubahan waktu pengerasan alginat akibat perbedaan suhu air serta mengetahui hubungan antara suhu air dan waktu pengerasan. **Metode:** Tujuh kelompok adonan alginat yang dipersiapkan dengan mencampur bubuk alginat dan air, masing-masing menggunakan suhu antara 13° C–28° C dengan interval 2,5° C. Pengukuran waktu pengerasan alginat dilakukan sesuai dengan spesifikasi ADA no.18. Sebuah cetakan sampel terbuat dari pralon berbentuk cincin ($\Theta = 30$ mm, $t = 16$ mm) ditempatkan di atas plat datar dan dipenuhi dengan adonan alginat. Pengukuran waktu pengerasan dilakukan segera dengan menyentuh ujung datar batang akrilik yang telah dipoles di permukaan adonan alginat. Waktu pengerasan alginat diukur dari awal pencampuran bubuk alginat dan air sampai dengan waktu awal ketika alginat tidak melekat di ujung batang. Data waktu pengerasan yang diperoleh dianalisis menggunakan uji statistik One-way Anova, LSD dan Pearson. **Hasil:** Suhu air antara 13° C–28° C telah menghasilkan waktu-waktu pengerasan alginat 87 detik hingga 119,4 detik yang berbeda signifikan ($p < 0,01$). Waktu pengerasan antar grup juga menunjukkan perbedaan signifikan ($p < 0,01$). Antara suhu air dan waktu pengerasan alginat terdapat hubungan terbalik ($r = -0,968$). **Kesimpulan:** Suhu air antara 13° C–28° C dengan interval 2,5° C menghasilkan perbedaan waktu pengerasan alginat; makin rendah suhu air yang digunakan untuk mencampur makin panjang waktu pengerasan alginat.

Kata kunci: Alginat, suhu air, waktu pengerasan, polimerisasi

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INTRODUCTION

Alginates are the most commonly used impression material to reproduce teeth and its surrounding tissue. Popularity of alginate is related to its ability to reproduce detailed anatomical impression of teeth and its surrounding tissue, when it is manipulated well.^{1,2} Internship students in the teaching hospital are not yet able to manipulate alginate well and thus not yet able to produce alginate dough with good consistency; whereas, students are expected to produce detailed and complete impression of teeth and other tissues. Alginate dough produced by the students are often set too fast when used to make impression in the mouth, or even set far before it is used to make impression. A study showed that room temperature without air conditioning, ranging from 24°C–31°C, contributed to alginate's setting time.³ In such condition, students often need to reduce the amount of alginate powder or add more water than what is recommended by the factory to delay alginate setting time. These methods will reduce mechanical strength of the alginate impression.^{4,5}

Alginates are salts from alginic acid that is obtained from brown algae. Alginic acid is part of organic polymers from polysaccharide family, composed of two units β -L-guluronate monomers (G unit) and α -D-manuronate (M unit) with combination of [GM-MG]_n.⁶ processed alginates contain cation, e.g. K²⁺, Na⁺, Ca²⁺, which is used as primary components in alginate impression material. Apart from that, alginate impression material also contains other components, i.e. calcium sulfate salts (CaSO₄), monovalent retarder natrium triphosphate (Na₃PO₄) and potassium alginic (K₂nAlginic). All components take part in the setting process of alginates.⁷

The setting process of alginates contains of series of reaction. The first reaction series, polymerization and gelation, may be influenced by the size of alginates' particles and/or retarder concentrations. Manipulating retarder concentrations are the most reliable and controlled method to delay the setting time of alginates.^{7,8} Such method can only be done during the fabrication of alginate powders. The easiest method to delay the setting time of alginates and prolong manipulating time is to use slow setting type of alginates; such type provides prolonged setting time than the normal one.⁷ If such type is not available, the normal one may still be used with water temperature lower than 80°F (\pm 28°C), as recommended by the manufacturer. The use of relatively low water temperature is important since it reduces collision among molecules in chemical reaction. The amount of the collision between substances' molecules reacted in time unit determines the rate of chemical reaction.⁹ The less collision will slower the chemical reaction and thus the completion of the chemical reaction will have longer time. In such case, the application of lower water temperature will enable longer setting time.

Previous research has studied about the influence of temperature on the setting time of alginate. The research studied water with 10°C difference of temperature to

mix alginates.¹⁰ Water temperature during the mixing of alginates is highly sensitive; even small difference of temperature may change the rate of the setting process of alginates.⁹ The present study has used water temperature difference lower than 10°C, i.e. 2.5°C, in order to obtain detailed impression results. Water temperature used were between 13°C to 28°C; this was because the laboratory temperature without air conditioning was approximately 28 (\pm 1)°C and the lowest temperature achieved by adding ice cubes was 13 (\pm 1)°C. Therefore, the study aimed to determine the changes in setting time due to differences in water temperature between 13°C to 28°C and to determine the correlation between water temperature and the setting time.

MATERIALS AND METHODS

Firstly, alginate dough was prepared according to its manufacture direction. A part (equivalent to 6.5 gram) of alginate powders with normal setting type (New Kromopan[®], Schultz Science Dental Product, Jerman, no. batch 015038011062) and water (equivalent to 17 ml) were put in a plastic bowl, then mixed and pushed with spatula to the wall of the bowl to get a homogenous dough. Water temperature ranged between 13°–28°C with 2.5°C interval; there were 7 groups with different water temperature to make the dough.

The test of alginate setting time followed ADA specification No. 18.¹¹ Specimen molds were made from plastic cylinder (pipe) with 30 mm in diameter and 16 mm in height placed in glass plate. Alginate dough in each group was poured into one specimen mold. The excess dough is removed using spatula until the surface of the dough reached the mold's height. A stick made from resin acrylic with 10 cm in length and 6 mm in diameter was placed in contact to the surface of the specimen. Alginate dough stucked to acrylic stick was cleaned and the contact was repeated with 10 seconds time interval. The setting time of alginate was counted from start of alginate mixing until no sticky dough found in the bar. The total number of specimens were 70.

Data of alginate setting time were analyzed with one-way ANOVA test and continued with LSD test and Pearsons test to find out the relationship between different temperature group and alginate setting time.

RESULTS

The setting time of alginate with water temperature between 13°–28°C was shown in the Table 1. It showed that water temperature between 13°C–28°C produced various setting time of alginate, ranging from 87 seconds to 119.4 seconds. The longest difference of alginate setting time was 8.8 seconds, produced by temperature 25.5°C and 28°C, while the shortest time difference was 3.8

Table 1. Setting time of alginate impression material with water temperature between 13° C-28° C

Water temperature (0C)	n	Setting time (second)
28.0	10	87.0 ± 1.83
25.5	10	95.2 ± 2.82
23.0	10	100.7 ± 2.56
20.5	10	107.6 ± 2.42
18.0	10	111.9 ± 2.81
15.5	10	115.7 ± 2.85
13.0	10	119.0 ± 2.92

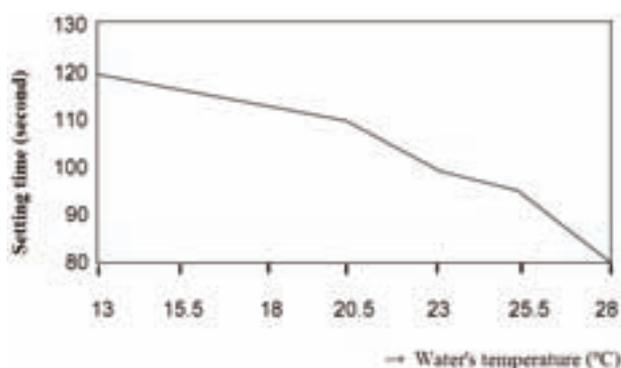


Figure 1. The setting time of alginate was longer in the application of water with lower temperature.

seconds, i.e. between 15.5° C and 18° C. Time difference in alginate setting time among seven groups ranged between 3–10%. The relationship between water temperature and alginate setting time was shown Figure 1.

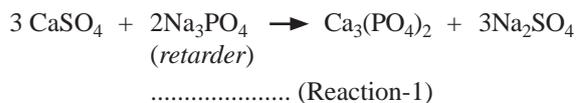
It showed that the shortest alginate setting time was at 28° C, while the longest was at 13° C (Figure 1). This showed that the relationship between water temperature and setting time was inversely proportional.

One way ANOVAtest showed that the setting time of alginates in different water temperature were significantly different (p<0.01). LSD test showed that the setting time between each temperature group were also significantly different (p<0.01). Pearson’s test proved that there was significantly inverse correlation between water temperature and the setting time (r = -0.968).

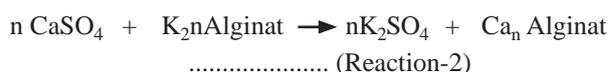
DISCUSSION

Longer set of alginate in the application of lower water temperature indicated that there was a delay in the setting time of alginate. Prolonged set of alginate has already started since alginate powder is wet with water until the alginate turns into irreversible hydrocolloid gel. Chemically, prolonged setting process may occur at the early stage of chemical reaction, polymerization stage and gelation stage. Early reaction that takes place since the

mixing of alginate powder with water triggers chemical reaction between CaSO₄ with Na₃PO₄ retarder in the alginate powder, as reaction-1 below:⁷



The reaction occurs continuously as long as there is retarder in reaction-1. Retarder maintains [GM-MG]n polymers to stay in water soluble form. In such case, reaction-1 delays the next reaction to take place. After all of retarder (Na₃PO₄) reacts in the reaction-1, then reaction-2 takes place as follow:⁷



Since there is no more retarder in reaction-2, CaSO₄ reacts with K₂nAlginic to produce Ca_nAlginic (calcium alginic/alginate). Physically, reaction-2 is the alginate polymerization, which is the merging of [GM-MG]n polymers into alginate that is cross-linked.⁷ This process is illustrated in Figure 2.

Cross-linked alginate occurs with the existence of CaSO₄ as the source of calcium ions (Ca⁺) to bind {GM-MG}n alginate.^{12,13} Ca⁺ ions make ether-oxygen bridge to unify carbon (C) at C1 position from M unit and C4 from G unit to become a cross-linked structure between [GM-MG]n polymers.

Beside delaying reaction-1 and reaction-2, the application of lower water temperature may also delay alginate gelation. At the early gelation, the alginate formed is still water soluble. Therefore, at the contact with acrylic bar, alginate still stuck to the bar. After passing the high

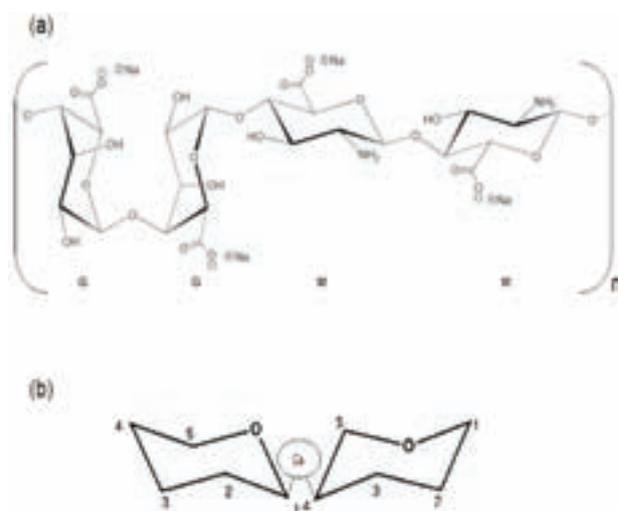


Figure 2. Scheme diagram (a) alginate expressed as Guluronat acid (G unit) Manuronat acid (M unit) forming (b) cross-linked at C1 and C4.¹²

viscosity solution stadium, alginate swells and eventually looks like gel-shaped thick paste.^{12,13} At the end of gelation stadium, alginat will no longer water soluble that can not be part. At this condition, alginate does not stick in the acrylic bar.

During the setting process, water temperature influences the efficiency of the collision between molecules in chemical reaction through kinetic effectivity.⁹ The application of higher water temperature gives increasing effect on the efficiency of the collision between reacted molecules and thus produces an effective molecular collision kinetics. On the other hand, the application of lower water temperature produces less effective molecular collision kinetics. Manipulating alginate using water temperature close to 13° C, produces kinetic effectivity of these collision and thus produces prolonged setting time of alginate. In other words, the lower water temperature used to mix the alginate powder, the longer alginate setting time produced.

Alginate setting time in this study could be compared to the previous studies. Referring to ADA,¹⁰ alginate setting time in normal setting type alginate (with water temperature $\pm 18^\circ\text{C}$) is ranging from 120 to 390 seconds, other studies,⁹ 102 to 282 seconds, while this study's result is 87 to 119.4 seconds. Even though the two studies using normal setting type impression material, it is likely that the time differences were contributed by room temperature and/or retarder concentration contained in the alginate. Retarder concentration may vary in every alginate with different trade name, as what is used in the three studies above.

Alginate setting time obtained from this study may be useful for students during alginate manipulation. Students may choose different water temperature based on their skill and competence to prolong setting time. Therefore, less skilled students in impression technique with requirement to produce good alginate impression can still use normal setting alginate type by adjusting water temperature used to mix the alginate.

This study showed that water temperature between 13° C to 28° C with a interval of 2.5° C produced significant

differences in alginate setting time; the lower the water temperature used, the longer the setting time produced.

REFERENCES

1. Nandini VV, Venkatesh KV, Nair C. Alginat impression: A practical perspective. *J Conserv Dent* 2008; 11(1): 37–41.
2. Ashley M, McCullagh A, Sweet C. Making a good impression: A 'how to' paper on dental alginate. *Dent Update* 2005; 32: 169–75.
3. Irnawati D, Sunarintyas S. Functional relationship of room temperature and setting time of alginate impression material. *Dent J (Majalah Kedokteran Gigi)* 2009; 42(3): 137–40.
4. Nallamuthu NA, Braden M, Patel MP. Some aspects of the formulation of alginate dental impression materials-setting characteristics and mechanical properties. *Dent Mater* 2012; 28(7): 756–62.
5. Woortman R, Kleverlaan CJ, Ippel D, Feilzer AJ, Cavex Holland BV. The effect of mixing method on the properties of alginate. Scientific Group Programme. Annual Meeting of IADR Continental European and Israeli Divisions 2007.
6. Murillo-Alvarez JI, Hernandez-Carmora G. Monomer composition and sequence of sodium alginate extracted at pilot plant scale from three commercially important seaweeds from Mexico *J Appl Phycol* 2007; 19: 545–8.
7. Anusavice KJ. Phillips' science of dental materials. 11th ed. St. Louis: Elsevier; 2003. p. 205-9, 231–48.
8. Lemon JC, Okay DJ, Powers JM, Martin JW, Chambers MS. Facial moulage: the effect of a retarder on compressive strength and working and setting times of irreversible hydrocolloid impression material. *J Prosthet Dent*. 2003; 90(3): 276–81.
9. Wright M. Theories of chemical reaction-collision theory in an introduction to chemical kinnetics. John Wiley & Sons, Ltd; 2004. p. 99–110.
10. Rianti D. Pengaruh variasi suhu air saat pencampuran bahan cetak alginat terhadap waktu pengerasan dan ketepatan model kerja yang dihasilkan. *Majalah Kedokteran Gigi (Dent J)* 1998; 31 (4): 115–8.
11. Barr JH, Bowen R. ADA specification no. 18 - impression materials. In: *Guide to Dental Materials and Device*. 7th ed. Chicago: American Dental Association; 1975. p. 219–23.
12. Draget KI, Smidsroed O, Skjak-Braek G. Alginates from Algae. In: Steinbuechel A, Rho SK, eds. *Polysaccharides, polyamides in the food industry - properties, production and patents*. Wiley-VCH Verlag GmVH & Co Weinbeim; 2006. p. 379–82.
13. Siew CK, Williams P, Young NW. New Insights into the mechanism of gelation of alginate and pectin: Charge annihilation and reversal mechanism. *Biomacromolecules*. 2005; 6(2): 963–9.