

Majalah Kedokteran Gigi

Dental Journal

(Majalah Kedokteran Gigi)

2021 December; 54(4): 195-199

Original article

The effects of mixing slurry water with type III gypsum on setting time, compressive strength and dimensional stability

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ABSTRACT

Background: Type III gypsum is a material used to make dental master casts. It may be added to an accelerator, such as slurry water, to shorten setting time. Calcium sulphate in slurry water may affect setting time, compressive strength and dimensional stability. **Purpose:** The study evaluated the effect of slurry water on the setting time, compressive strength and dimensional stability of type III gypsum. **Methods:** Eighty-one samples were made of type III gypsum, divided into three groups: group A was gypsum mixed with 1% slurry water, group B, gypsum mixed with 2% slurry water and group C, gypsum mixed with distilled water. Each sample was formed using a standardised master mould. For testing setting time, a cylindrical mould 25 mm in diameter and height was used; for compressive strength testing, the cylindrical mould was 20 mm in diameter and 40 mm in height; and for dimensional stability testing, a pair of cylindrical, ruled block and mould were used. Setting time was tested using Vicat's apparatus; compressive strength was tested using a universal testing machine; and dimensional stability was tested using digital callipers. The data were analysed by one-way analysis of variance (ANOVA) and least significant difference (LSD) tests. **Results:** One-way ANOVA and LSD tests showed significant differences in the effect of slurry water on the setting time, compressive strength and dimensional stability of type III gypsum.

Keywords: compressive strength; dimensional stability; setting time; slurry water; type III gypsum

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INTRODUCTION

Gypsum is a mineral widely found in nature, categorised as calcium sulphate dihydrate (CaSO₄.2H₂O). It is usually white to yellowish white and is found as a solid mass.¹⁻³ Gypsum products are widely used in dentistry for the production of study and master casts. To form a powder from mineral gypsum, a manufacturer heats the dihydrate, which cause it to lose water. It is then ground to produce a powdered hemihydrate (CaSO₄.¹/₂H₂O). When the hemihydrate is again mixed with water, it is converted back to a dihydrate and again becomes a solid mass.³ According to the specification no. 25 of the American Dental Association (ADA), dental gypsum products are classified into five types: type I, impression plaster; type II, model plaster; type III, dental stone; type IV, high strength dental stone; and type V, high strength, high expansion dental stone.¹⁻⁴

Type III gypsum, known as dental stone, is often used to produce a working cast (master cast) due to its high strength against fracture and abrasion. Dental stone is denser and stronger than plaster, because its particle characteristics require less water.³ The properties of type III gypsum are setting time, compressive strength, hardness and abrasion resistance, setting expansion, dimensional stability and detail reproduction.^{1–4} The working cast must have a specific setting time, strength, and dimensional stability over time, so as to guarantee the accuracy of the model – properties that are very important during the manufacturing process.

Based on the ADA specification no. 25, type III gypsum has a setting time of 8–16 minutes, compressive strength of 20.7–34.5 MPa and dimensional stability of 0–0.20%.³ Setting time, compressive strength, and dimensional stability values are affected by the addition of an accelerator.¹ The use of different types of water, such as slurry water and distilled water, can be used in the gypsum manipulation process. Several studies have identified that the addition of an accelerator can change gypsum's properties, due to differences in mineral content of different solutions.

Slurry water is an accelerator that contains calcium sulphate and is obtained by placing clean, completely set dental stone pieces in a plastic container of distilled water and allowing them to soak for 48 hours.⁵ Use of slurry water is a must.⁶ The calcium sulphate contained in slurry water acts as a catalyst that causes calcium sulphate dihydrate particles to form faster, thereby reducing the setting time when making a master cast. Thus, the aim of this study is to evaluate the effects of 1% and 2% slurry water on the setting time, compressive strength and dimensional stability of type III gypsum.

MATERIALS AND METHODS

This experimental research was carried out in a laboratory setting using a post-test-only control group design. The samples used were three different mixtures of type III gypsum, produced and divided into three groups: group A was type III gypsum mixed with 1% slurry water, group B, type III gypsum mixed with 2% slurry water and group C, type III gypsum mixed with distilled water. Each sample was formed using a standardised master mould according to ADA specifications: the master mould for setting time testing (ADA No. 25) was cylindrical and 25 mm in both diameter and height; for compressive strength testing (ADA No. 25) the cylindrical mould was 20 mm in diameter and 40 mm in height;⁷ and for stability dimension testing (ADA No. 19) a pair of cylindrical ruled block and gypsum mould with a 38 mm outer diameter, 30 mm inner diameter and 20 mm height.8

The number of samples in this study was calculated using the Federer formula, with nine samples for each of the three studied groups, totalling 27 samples. Each sample was then tested for setting time, compressive strength and dimensional stability – an overall total of 81 samples. The study was conducted in December 2020 at the Prosthodontics Laboratory, Faculty of Dentistry, University



Figure 1. 1% and 2% slurry water.

of Sumatera Utara and the Impact and Fracture Research Center, Laboratory of Mechanical Engineering, University of Sumatera Utara. The Research Ethics Commission at the University of Sumatera Utara approved the study (No: 832/ KEP/USU/2020), according to the Declaration of Helsinki on medical protocols and ethics.

The manufacture of slurry water began by mixing 100 g of type III gypsum powder with 30 ml of distilled water in a rubber bowl using a spatula for 60 seconds at a speed of about two revolutions per second until homogenous. The dough was then poured into a container and allowed to stand for 48 hours. Gypsum that had hardened was then crushed to form pieces. To make 1% slurry water, 1 g of gypsum pieces were soaked in 100 ml of distilled water for 48 hours (group A), and 2 g of gypsum pieces were soaked in 100 ml of distilled water for 48 hours, to make 2% slurry water (group B). Slurry water was stored at room temperature (Figure 1).⁹

To make the research sample, each master mould was first smeared with Vaseline, as evenly and as thinly as possible. The slurry water was shaken well before use, after which 30 ml of each solution (1%, 2% slurry water and distilled water) was weighed out using digital scales and placed in separate rubber bowls. Next, 100 g of type III gypsum powder, which had been weighed, was added gradually to each rubber bowl and stirred using a spatula for 60 seconds at a speed of approximately two revolutions per second until homogeneous. Once homogeneous, the gypsum mixture was poured slowly, with the help of a spatula, into the master mould, positioned on a glass slab that was vibrated for a few seconds until the mould was full. The excess dough was flattened using a glass slab placed on top of the master mould and pressed firmly until it touched the top surface of the master model.

Setting time was tested using Vicat's apparatus. The mould containing type III gypsum dough was placed under the measuring needle, which was 1 ± 0.005 mm in diameter and a plunger weighing 2.942 ± 0.005 N (300 ± 0.5 grams). The setting time was determined by bringing the tip of the needle into contact with the surface of the material. The needle was released and allowed to penetrate the sample at 15-second intervals. The needle was wiped clean after each penetration and the master mould moved to permit the next penetration in a new area. The setting time of the type III gypsum was calculated from the start of the mixing process until the time when the needle could no longer penetrate.^{1,7}

Compressive strength was tested after the samples had hardened completely for 24 hours, by applying a compressive load to each sample until it broke, using a universal testing machine. The data obtained at the failure point of each sample were recorded in kilogram-force (kgf), and the results of the type III gypsum compressive strength calculated in megapascals (MPa) by using the formula:⁷

Compressive Strength =
$$\frac{F}{A} = \frac{P \times 9.8}{\pi r^2}$$

Dental Journal (Majalah Kedokteran Gigi) p-ISSN: 1978-3728; e-ISSN: 2442-9740. Accredited No. 32a/E/KPT/2017. Open access under CC-BY-SA license. Available at https://e-journal.unair.ac.id/MKG/index DOI: 10.20473/j.djmkg.v54.i4.p195–199 where:

F = the force or load at point of failure (N)

A = cross-sectional surface area (mm^2)

P = compressive load (kgf)

Dimensional stability was evaluated 24 hours after making the samples, by measuring the length of the lines using digital callipers. The distance between the crosslines (cd - c'd') on lines A, B and C of each sample was measured. The measurements were then totalled, and the mean value obtained. Next, the data from the measurement of dimensional changes were converted into a percentage by using the formula:⁹

$$\frac{L_1 - L_0}{L_0} x \, 100\%$$

where:

 L_1 = the mean of line length obtained on the sample (mm) L_0 = the line length on the master mould (mm)

All the data were analysed using statistical tests run on the SPSS software program. The mean and standard deviation of the data were obtained using descriptive analysis. The effect of mixing slurry water with type III gypsum on setting time, compressive strength and dimensional stability were evaluated using a one-way analysis of variance (ANOVA). The differences between the studied groups were compared using the least significant difference (LSD) post hoc test. To use one-way ANOVA, the data obtained from the study must be normally distributed. To establish whether the data were normally distributed or not, a normality test was carried out using Shapiro–Wilk test (p>0.05).

RESULTS

Table 1 shows the mean and standard deviation of setting time, compressive strength and dimensional stability of type III gypsum mixed with each of the three different solutions. Statistical analysis using the Shapiro–Wilk test established that the data were normally distributed (p>0.05), following which a one-way ANOVA was used to evaluate the effects of mixing slurry water with type III gypsum on setting time, compressive strength and dimensional stability.

The ANOVA test showed a statistically significant difference (p<0.05) in the effect of mixing slurry water with type III gypsum on setting time, compressive strength and dimensional stability (Table 2). The LSD multiple comparison test showed a significant difference (p<0.05) between all the studied groups (Table 3).

DISCUSSION

In this study, type III gypsum powder was mixed with three different types of solutions, namely 1% slurry water, 2% slurry water and distilled water as a control. Slurry water contains minerals, unlike distilled water. Because the solutions used have different mineral compositions, this can affect the setting time, compressive strength and dimensional stability of type III gypsum.

The mean and standard deviation of setting time in group C was the longest relative to groups A and B.

 Table 1.
 Mean and standard deviation of setting time, compressive strength and dimensional stability of type III gypsum mixed with 1% slurry water (A), 2% slurry water (B) and distilled water (C)

| Group | Setting time (s) | | Compressive strength (MPa) | | Dimensional | stability (%) |
|-------|------------------|-------|----------------------------|------|-------------|---------------|
| | Mean | SD | Mean | SD | Mean | SD |
| А | 421.67 | 31.25 | 22.37 | 0.59 | 0.088 | 0.028 |
| В | 346.89 | 22.61 | 20.07 | 0.57 | 0.130 | 0.032 |
| С | 596.22 | 57.82 | 25.87 | 2.00 | 0.053 | 0.028 |

Table 2. One-way ANOVA results of setting time, compressive strength and dimensional stability of type III gypsum mixed with1% slurry water, 2% slurry water and distilled water

| Variable | Ν | f-value | p-value |
|-----------------------|----|---------|---------|
| Setting time | 27 | 91.50 | 0.0001 |
| Compressive strength | 27 | 49.33 | 0.0001 |
| Dimensional stability | 27 | 15.35 | 0.0001 |

Table 3. Least Significant Difference results of setting time, compressive strength and dimensional stability of type III gypsum mixed with 1% slurry water (A), 2% slurry water (B) and distilled water (C)

| Group | | p-value | | | | |
|-------|---|--------------|----------------------|-----------------------|--|--|
| | | Setting time | Compressive strength | Dimensional stability | | |
| Α | В | 0.001 | 0.001 | 0.006 | | |
| А | С | 0.000 | 0.000 | 0.020 | | |
| В | С | 0.000 | 0.000 | 0.000 | | |

Dental Journal (Majalah Kedokteran Gigi) p-ISSN: 1978-3728; e-ISSN: 2442-9740. Accredited No. 32a/E/KPT/2017. Open access under CC-BY-SA license. Available at https://e-journal.unair.ac.id/MKG/index DOI: 10.20473/j.djmkg.v54.i4.p195–199 The type III gypsum, when mixed with 1% and 2% slurry water, had shorter setting times than when mixed with distilled water, due to the presence of calcium sulphate. The results of this study also showed that the type III gypsum mixed with 2% slurry water group had a shorter setting time than the type III gypsum group mixed with 1% slurry water, due to the respective differences in the amount of calcium sulphate contained in 1% and 2% slurry water. Calcium sulphate dihydrate particles act as the core of the crystallisation process. The greater the number of calcium sulphate dihydrate particles, the greater the crystallisation formation.^{3,10} The presence of a sufficient number of crystallisation nuclei in the slurry water can accelerate the crystallisation process and the solubility of calcium sulphate hemihydrate to become dihydrate, with the result that the setting time of type III gypsum becomes faster. The results of this study are in line with previous research conducted by Denizoglu et al.,¹¹ which compared the setting times of type III gypsum mixed with distilled water, tap water, 2% and 16% slurry water and found that the use of slurry water shortened type III gypsum setting time. The mean setting time of type III gypsum mixed with 16% slurry water was shorter than that of the type III gypsum mixed with 2% slurry water.11

The results of the study for the compressive strength variable showed that the type III gypsum mixed with distilled water group had the highest compressive strength value compared to the gypsum type III groups mixed with 1% and 2% slurry water. The results of this study are in line with research conducted by Dewi,⁹ which found that use of distilled water had the highest compressive strength value when compared to the use of tap water and slurry water. The addition of slurry water, which acts as an accelerator, can reduce the compressive strength of gypsum, due to the crystal shape becoming irregular, which reduces intracrystalline cohesion.⁹ Furthermore, the compressive strength of gypsum is closely related to its surface hardness. The higher the surface hardness value of gypsum, the higher its compressive strength value. Ayoub et al.,¹² found that the greatest type IV gypsum surface hardness was obtained in samples mixed with distilled water, while the least gypsum surface hardness was obtained in samples mixed with slurry water.

Different mineral content between slurry water, both 1% and 2%, and distilled water affects the value of the compressive strength of type III gypsum. Musa et al.,¹³ showed that use of distilled water produced the greatest compressive strength value compared to five other treatment groups, namely tap water, tap water mixed with gypsum powder, slurry water made with tap water, distilled water mixed with powder gypsum and slurry water made with distilled water. The gypsum group mixed with slurry water had a lower compressive strength than the group using distilled water. This may be related to the absence of any mineral content in the distilled water, such that the crystal form is regular, relatively non-porous and denser. The mineral contained in the slurry water, namely calcium

sulphate, may reduce intracrystalline cohesion, resulting in a reduction in the gypsum compressive strength. Mixing slurry water with type III gypsum reduces the compressive strength, because calcium sulphate particles cause the crystals to become irregular in shape, which affects the ability of gypsum crystals to grow and causes pores, resulting in a more brittle gypsum product.¹³ Vyas et al.,¹⁴ found that, generally, the gypsum group with sulphate additives had a lower resistance to compressive strength than the control group without additives. Incorporating additives may cause an increase in the concentration of additives in the gypsum dough, such that the number of gypsum crystals formed from the overall volume decreases, with a corresponding decrease in intracrystalline cohesion, resulting in gypsum products with a low compressive strength. The function of additives is to increase the reaction rate, so it is possible that the reaction occurs so fast that some hemihydrate crystals are not completely formed into dihydrates, which causes an increase in hemihydrate crystals, producing a weak gypsum product.14

The results of the study for the dimensional stability variable found that the type III gypsum mixed with distilled water group had the lowest dimensional stability value compared to the groups of type III gypsum mixed with 1% and 2% slurry water. This is related to the different mineral content between distilled water and slurry water. The results of this study are in line with research conducted by Dewi,⁹ who found that the use of distilled water had the smallest dimensional change value when compared to the use of tap water or slurry water.

The dimensional stability of gypsum is generally closely related to its setting expansion. The higher the gypsum expansion value, the higher the gypsum dimension change value. Denizoglu et al.,¹¹ found that mixing gypsum with 2% and 16% slurry water increased the expansion value in the first 24 hours. 16% slurry water had the greatest expansion value when compared to distilled water and 2% slurry water. This may be caused by the presence of calcium sulphate in the slurry water, which acts as a nucleation core for the growth of calcium sulphate dihydrate crystals, resulting in an increased number of dihydrate crystals, which causes greater overlapping of crystals and a greater setting expansion.¹¹

The value of dimensional stability of gypsum mixed with slurry water is greater than that of the group of gypsum mixed with distilled water. This may be due to the relatively lower water content of slurry water, which causes a greater setting expansion. This reduced water content results from an increase in calcium sulphate particles, which attract water particles during the gypsum setting process, with a resulting lower water content.¹⁰ Alberto et al.,¹⁵ noted that water content may affect the setting expansion of gypsum. The lower the water content at the time of mixing, the greater the setting expansion of gypsum. The water content in gypsum affects both the internal growth of the dihydrate crystals and the protusion of the dihydrate crystals.¹⁵ From the data analysis and discussion, the study concludes that

the use of slurry water, both 1% and 2%, may shorten setting time, decrease compressive strength and increase dimensional change of type III gypsum. Further analysis of the effects of slurry water on other properties of type III gypsum is needed.

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