Characteristics of a new dental stone mixed by shaking

Yuko NAGASAWA, Yasushi HIBINO, Hirotaka SHIGETA, Yoshikazu EDA, Shigeki MATSUMOTO and Hiroshi NAKAJIMA

Division of Dental Biomaterials Science, Department of Restorative and Biomaterials Sciences, Meikai University School of Dentistry, 1-1 Keyakidai, Sakado, Saitama 350-0283, JAPAN
Corresponding author, Yuko NAGASAWA; E-mail: nagasawa@dent.meikai.ac.jp

This study evaluated the physical and mechanical properties of a dental stone mixed by shaking. A shake-mix dental stone (Shake! Mix STONE; SM) was characterized in comparison with three conventional dental stones. The fluidity at pouring time, setting time, density, powder particle distributions, linear setting expansion, compressive strength and surface reproduction of detail for dental stones were investigated. The marginal adaptations of cast crowns to dies made with each stone were also determined. SM had higher fluidity and faster setting time than the other stones (p<0.05). The setting expansion of SM at 2 h was lower than those of other two stones (p<0.05). The 15-min compressive strength of SM was higher than the others (p<0.05). There were no significant differences in the marginal adaptations of the cast crowns fabricated using all the stones (p>0.05). In spite of the different mixing method, the shake-mix type dental stone had comparable physical and mechanical properties to the conventional dental stones.

Keywords: Dental stone, Shake mixing, Setting time, Setting expansion, Strength

INTRODUCTION

Gypsum —calcium sulfate dihydrate— is an abundant natural mineral resource. In dentistry, gypsum or plaster is provided to dental offices or dental laboratories in the form of calcium sulfate hemihydrate, which is synthesized chemically or produced from natural gypsum. Plaster has been used as a material for casting since 1756 and for impressions since 1844. In addition to making casts and impressions, plaster is widely utilized in various procedures in dental works. Furthermore, calcium sulfate, the main component of gypsum, is added as a setting agent in the alginate impression materials frequently used in dental treatments. Plaster is therefore an essential material for dental treatments and dental laboratory procedures.

In current dental works, plaster or stone is mainly used for casts or models. The study casts (models) are usually made from beta-calcium sulfate hemihydrate, and the working casts (models) are made from alpha-calcium sulfate hemihydrate. The powder of calcium sulfate hemihydrate reacts with water to form calcium sulfate dihydrate, and then sets. This setting reaction is known to be affected by the water/powder mixing ratio, mixing conditions and addition of chemicals as setting accelerants or retardants. Several investigators have reported the effects of mixing conditions on the physical properties of dental stones. Earnshaw and Smith found that the diametral tensile strengths of stones was less affected by changes in water/powder mixing ratios than the compressive strength, and that there was no significant difference in either diametral tensile strength or compressive strength of stone between mechanical mixing and hand mixing procedures. Jørgensen and Kono reported the relationships of the mixing procedures, the porosity in stone and the compressive strength of stone. They found that the porosity of stone was reduced from 1.7% by using mixing without a vacuum to 0.6% by using vacuum mixing, and that vacuum mixing increased the compressive strength of stone at decreased the water/powder mixing ratio.

In 2008, Azer et al. investigated the compressive strength, diametral tensile strength, surface porosity, microhardness, setting time, and setting expansion of four different stones. One of the stones (HandiMix) examined in their study was recommended by the manufacturer to be mixed by shaking. They reported that the mixing procedures of stones did not affect the physical properties of stones and that the properties of stone mixed by shaking were comparable to those mixed by the ordinary mixing procedure.

Although a stone mixed by shaking was reported by Azer et al., to the best of our knowledge, no further studies on stones mixed by shaking have been reported during the past decade. Recently, a Type 4 stone mixed by shaking (Shake! Mix STONE) was newly developed and is available on the market. This stone is mixed in a specially-made plastic container with water by hand-shaking. However, no studies on the properties of this new dental stone mixed by shaking have been reported to date.

The purpose of this study was to characterize the physical and mechanical properties of Shake! Mix STONE and the fitting of cast crowns fabricated using this stone, to discuss its applicability in dental works. The study hypothesized that the stone mixed by shaking exhibits properties comparable to those of the conventional stones mixed by the ordinary procedure.
MATERIALS AND METHODS
A new dental stone mixed by shaking and three conventional dental stones mixed using a rubber bowl and spatula were investigated in this study. The water/powder ratios used for each stone in this study are listed in Table 1.

The shake-mix type dental stone (SM) was mixed in its specially-made plastic cylindrical container. The measured water was placed in the container, then the weighed powder was added and the lid was closed quickly. Immediately after closing the lid, the container was reciprocated by hand horizontally and linearly in the long axis direction of the container for 20 s at 3 reciprocations per second. For the other three conventional dental stones, after measuring, the powder and water were mixed with standard procedures by hand using a rubber bowl and metal spatula for 30 s at 120 revolutions/min. All dental stones were mixed in accordance with the respective manufacturers’ recommendations. Deionized water was used.

After the mixing the stones, the following properties were examined as outlined in ISO 6873, except for marginal adaptation. The surfaces of set stones were observed using a scanning electron microscope (SEM). All examinations were carried out at 23±2°C. In order to characterize the stone powders, the powder particle size analysis was made by laser diffraction and the powder particles morphology was examined using SEM.

The results were statistically analyzed using ANOVA/Scheffé’s test at α=0.05.

**Powder particle size analysis and powder particles morphology**
The particle sizes distributions of each dental stone were investigated according to ISO 13320 using the laser particle size analyzer (LA-960, HORIBA, Kyoto, Japan). The measurements were repeated for fifteen times for each dental stone.

The powder particles of each dental stone (calcium sulfate hemihydrate crystals) were observed by SEM (JSM-6360LV, JEOL, Tokyo, Japan) after the gold spattering (20 nm thick) at 20 kV of acceleration voltage.

**Fluidity at pouring time**
The fluidity at pouring time was determined according to ISO 6873. A cylindrical metal mold (35 mm in diameter, 50 mm high) was placed on a glass plate. The mold was completely filled and the mixed material levelled-off so that it was flush with the top of the mold. The cylindrical metal mold was lifted vertically from the glass plate at a rate of approximately 10 mm/s and the mix allowed to slump or spread over the plate. One minute after the elevation of the metal mold, the major diameter and minor diameter of the stone specimens were recorded, and the mean of these was taken as the fluidity for the sample. Fluidities at 45, 60, 75, 90, 105 or 120 s after the start of mixing were measured. Five specimens were prepared and examined for each stone.

**Setting time**
Cylindrical specimens (20 mm in diameter, 30 mm high) of each dental stone were prepared using metal molds according to the respective manufacturer’s recommended mixing ratios. The setting times of dental stones were determined using a penetrometer needle (needle length 50 mm, needle dia. 1.00±0.05 mm and weight 300±1 g), as outlined in ISO 6873. The setting time was recorded as the total time from the start of mixing to the time when the needle first failed to penetrate the specimen to a depth of 2 mm. Five specimens were prepared and tested for each stone.

**Density**
Cylindrical specimens (25 mm in diameter, 30 mm high) of each dental stone were prepared using plastic molds at room temperature. Thirty minutes after the start of mixing, the specimens were removed from the mold and weighed using the weight balance (ME204E, METTLER TOLEDO, Columbus, OH, USA) as a baseline data. The specimens were stored in dry condition (approximately 20 % relative humidity) at 37±2°C. The specimen weights were determined again at 60 min after the start of mixing. The density (apparent density) of the specimens of each dental stone was calculated by the weight of each specimen divided by the volume of each specimen. Five specimens were prepared and tested for each stone.

**Linear setting expansion**
The linear setting expansion was determined using an extension meter for measuring setting expansion with a square cross-section trough (20-mm width, 20-mm depth and 100-mm length), as outlined in ISO 6873. Dental stone mixture was poured into the square metal mold, to

| Material                | Manufacturer                  | Lot No.  | W/P ratio | Code |
|-------------------------|-------------------------------|----------|------------|------|
| Shake! Mix STONE        | GC, Tokyo, Japan              | 1703281  | 0.25*      | SM   |
| NEW PLASTONE II         | GC                            | 1707071  | 0.23*      | NP   |
| HYDRO GIPS              | SHOFU, Kyoto, Japan           | 061772   | 0.28*      | HG   |
| Dental Stone            | Ransom & Randolph Dentsply, Maumee, OH, USA | 100217101 | 0.30*      | DS   |

*manufacturer’s recommended mixing ratio
contact with a dial gauge. Sixty seconds before the setting time obtained from the setting time measurement, the dial gauge reading was recorded as the initial reading (A), and reading values were continuously recorded up to 2 h. Two hours after the start of mixing, the dial reading was recorded as the final reading (B). The difference in readings between A and B divided by the initial length of the stone specimen was calculated as the linear setting expansion for each specimen. Five specimens were prepared and tested for each stone.

Compressive strength
The compressive strength was determined as outlined in ISO 68736). Cylindrical specimens (20 mm in diameter, 40 mm high) of each dental stone were prepared using metal molds. Each specimen was stored in air at 23±2°C for up to 60 min. The compressive strength of each dental stone was determined using a universal testing machine (AG-50kNG, AUTOGRAPH, SHIMADZU, Kyoto, Japan) with a loading speed of 5±2 kN/min at 15, 30, 45 and 60 min after the start of mixing. Five specimens were prepared and tested for each stone.

Reproduction of detail
The surface reproduction of each dental stone was investigated in accordance with ISO 68736). Figure 1 shows the test block made from stainless steel used in this examination. The test block has 4 notches with different line widths as follows: groove a: 50±8 µm, groove b: 20±4 µm, groove c: 75±8 µm, groove d: 75±8 µm. A polyvinylsiloxane impression material (EXAMIXFINE INJECTION TYPE, Lot. 1703161, GC, Tokyo, Japan) was used to make impressions. After removal of the impression from the test block, each dental stone was mixed at the respective manufacturer’s recommended powder/water ratio and poured into the impression to prepare a stone cast. The reproducibility of the lines on the model surface was observed and assessed using a
stereomicroscope (×10, DSZT44, Carton, Tokyo, Japan).

**Marginal adaptation of the cast crown**

Five master dies (full crown type, 9 mm in diameter and 5 mm high, shoulder width 1 mm) made from epoxy resin were used in this study (Fig. 2). Impressions were made four times with each master die using vinyl polysiloxane impression material (EXAMIXFINE PUTTY TYPE, Lot. 1802131, GC, EXAMIXFINE INJECTION TYPE, Lot. 1703161, GC). Each dental stone was poured into each impression to fabricate a working cast according to the manufacturer’s instructions. This procedure was repeated four times for each of the master dies.

A wax pattern was prepared from each stone model and a cast crown made from Ag-Pd-Au alloy (Kinpara G12, Ag: 48.2%, Pd: 20.0%, Cu: 17.7%, Au: 12.0 % and others, Lot. 3003048, Ishifuku, Tokyo, Japan) was fabricated by the ordinary casting procedures. Vacuum mixing of the investment material (IDEAVEST MICRO, Lot. 1711101, GC) was carried out prior to investing the wax patterns.

The fitting of marginal regions of cast crowns to their master dies was determined by measuring the gap distance at the margins between the master die and the cast crown using a measuring microscope (×100, STM-5, Olympus, Tokyo, Japan). Four margin locations for each cast crown were measured, and the gap distances measured were averaged for each cast crown.

**SEM surface observation of set dental stones**

Cylindrical specimens of each stone material were prepared and kept in air at 23±2°C for 72 h after mixing. The specimens were coated with gold film (20 nm thick) and observed by SEM (JSM-6360LV, JEOL) at 20 kV of acceleration voltage.

## RESULTS

**Powder particle size analysis and powder particle morphology**

1. **Powder particle distribution**

   Figure 3 shows the particle size distributions of each dental stone. The powder particle size distributions revealed trimodal patterns in all dental stones. The measured particle sizes at 10 vol% (D10), 50 vol% (D50) and 90 vol% (D90) (µm, mean±SD) of the dental stones are shown in Table 2. ANOVA (Table 3) indicated that significant differences were found in powder particle size distributions among the dental stones tested, at D10, D50 or D90 (F=34.470, p<0.001 at D10, F=213.163, p<0.001 at D50 and F=47.788, p<0.001 at D90).

![Fig. 3](image_url)

**Table 2**  Powder particle sizes of each dental stone used in this study

| Material | D10 (µm)     | D50 (µm)     | D90 (µm)     |
|----------|--------------|--------------|--------------|
| SM       | 2.81±0.29    | 20.95±1.92   | 86.96±6.72   |
| NP       | 2.50±0.13    | 18.74±0.86   | 82.61±7.31   |
| HG       | 1.99±0.22    | 10.35±0.82   | 69.26±11.00  |
| DS       | 2.67±0.26    | 13.42±0.85   | 69.07±8.88   |

D10, D50 and D90 mean the particle sizes at 10, 50 and 90 vol%, respectively.
Table 3  Summary of analysis of variance for the particle sizes at D_{10}, D_{50} and D_{90}

| Source       | df | Sum of squares | Mean square | F     | p    |
|--------------|----|----------------|-------------|-------|------|
| D_{10}       |    |                |             |       |      |
| Dental stone | 3  | 5.826          | 1.942       | 34.470| <0.001|
| Error        | 56 | 3.155          | 0.056       |       | —    |
| Total        | 59 | 8.981          | —           | —     | —    |
| D_{50}       |    |                |             |       |      |
| Dental stone | 3  | 1052.192       | 350.731     | 213.163| <0.001|
| Error        | 56 | 92.141         | 1.645       |       | —    |
| Total        | 59 | 1144.333       | —           | —     | —    |
| D_{90}       |    |                |             |       |      |
| Dental stone | 3  | 3757.974       | 1252.658    | 16.712| <0.001|
| Error        | 56 | 4197.581       | 74.957      |       | —    |
| Total        | 59 | 7955.554       | —           | —     | —    |

D_{10}, D_{50} and D_{90} mean the particle sizes at 10, 50 and 90 vol%, respectively.

Fig. 4  SEM observation of powder of each dental stone.

Fig. 5 Fluidity at pouring time of dental stones examined in this study. Because the fluidity of SM was not able to be measured at 120 s, the data point for SM at 120 s was omitted.

2. Powder particle morphology

Figure 4 shows representative SEM photographs of particles of dental stones.

The crystal shapes of powder particles (calcium sulfate hemihydrate) of dental stones were hexagonal cylinder, cube-shaped particles or irregular in shape. Although SM appeared rather greater particle sizes than other stone powders, the powder morphologies were found to be similar among the dental stones used in this study.

Fluidity at pouring time

The measured fluidity (mm, mean±SD) of the dental stones is shown in Fig. 5. The fluidity of SM could not be obtained at 120 s because this stone was not able to spread out on the glass plate due to its rapid setting behavior. In order to compare the fluidity values among all the stones, the present study carried out the statistical analysis for fluidity data up to 105 s where the fluidity values of all stones were measureable. Two-way ANOVA
(Table 4) indicated significant differences in fluidity among the dental stones tested (F=1,910.453, p<0.001). A significant effect of the elapsed time after start of mixing was found in the fluidity (F=963.534, p<0.001). In addition, a significant interaction effect between the dental stones and times was found in fluidity results (F=741.089, p<0.001).

1. Effect of the elapsed time after dental stone mixing on fluidity
SM showed significantly greater fluidity at 45 s than at the other time periods (p<0.05). The fluidity of SM then decreased with time until 90 s (p<0.05), but no significant changes in fluidity were observed thereafter (p>0.05). There were no significant changes in fluidity for NP with time elapsed (p>0.05) from 45 to 105 s. The fluidity of HG at 45 s was significantly greater than at the other time periods (p<0.05) and decreased with time. HG revealed lower fluidity at 105 s than at 60 and 75 s (p<0.05). The fluidity of DS at 45 s was significantly greater than at the other time periods (p<0.05), but there were no significant differences in fluidity after 60 s (p>0.05).

2. Comparison of the fluidity among dental stones at each measurement time
Although the fluidity of SM decreased with time, those at 45, 60 and 75 s exhibited the greatest fluidity among all the dental stones tested (p<0.05). The fluidity of NP showed the second-highest values between 45 and 75 s (p<0.05), and had the highest values among the stones at 90 s and thereafter. Interestingly, SM had the lowest fluidity values at 90 and 105 s among all the dental stones examined.

No significant differences in fluidity values were observed at all time periods between HG and DS (p>0.05).

Setting time
Figure 6 shows the setting times (s, mean±SD) of the dental stones examined. Significant differences in setting time were found among the dental stones (F=6,319.613, p<0.001) (Table 5). SM revealed the shortest setting time (135±3 s), and NP the longest setting time (709±5 s) among the stones.

Density
The measured density (g/cm³, mean±SD) of the dental stones is shown in Table 6. Two-way ANOVA (Table 7) indicated significant differences in density among the
Table 7  Summary of analysis of variance for density

| Source      | df | Sum of squares | Mean square | F      | p   |
|-------------|----|----------------|-------------|--------|-----|
| Dental stone (A) | 3  | 0.088          | 0.029       | 124.194 | <0.001 |
| Time (B)    | 1  | 0.008          | 0.008       | 35.457 | <0.001 |
| A×B         | 3  | 0.001          | 0.000       | 0.743  | 0.534 |
| Error       | 32 | 0.008          | 0.000       |        |      |
| Total       | 39 | 0.104          | —           | —      | —    |

Table 8  Summary of analysis of variance for linear setting expansion

| Source      | df | Sum of squares | Mean square | F      | p   |
|-------------|----|----------------|-------------|--------|-----|
| Dental stone | 3  | 0.058          | 0.019       | 1,065.939 | <0.001 |
| Error       | 16 | 0.000          | 1.815×10⁻⁵ | —      | —    |
| Total       | 19 | 0.058          | —           | —      | —    |

Fig. 7 Linear setting expansion of dental stones used in this study.

dental stones tested (F=124.194, p<0.001). A significant effect of the elapsed time after start of mixing was found in density (F=35.457, p<0.001). At 30 min, there were significant differences in density among the dental stones (p<0.05). Except for between SM and NP, significant differences in density were observed among the dental stones at 60 min after the start of mixing (p<0.05). The values of density were significantly changed with time for all dental stones (p<0.05).

Linear setting expansion

Figure 7 shows the values of linear setting expansion for each dental stone, and the changes in linear setting expansion of the dental stones with time are shown in Fig. 8. There were significant differences in the values of linear setting expansion among the stones (F=1,065.939, p<0.001, Table 8).

SM, HG and DS but not NP exhibited increasing expansion rapidly up to approximately 20 min, and gradually thereafter. For NP, the rapid increase in expansion was prolonged to approximately 30 min after the start of mixing. Significant differences in linear setting expansion were observed among the dental stones (p<0.05). DS showed the lowest and HG the greatest linear setting expansion among the dental stones (p<0.05). SM showed less linear setting expansion than HG and NP (p<0.05).

Compressive strength

Changes in compressive strength (MPa, mean±SD) of the dental stones with time are shown in Fig. 9. Table 9 summarizes the results of two-way ANOVA for compressive strength measurements. Two-way ANOVA indicated significant differences in compressive strength among the dental stones tested (F=86.173, p<0.001). A significant effect of the elapsed time period after the start of mixing was found in the compressive strength (F=253.216, p<0.001). In addition, a significant interaction effect between the dental stones and times was found in compressive strength results (F=24.958, p<0.001).

1. Effect of the elapsed time after dental stone mixing

The compressive strength of SM was 31.9±3.5 MPa at 15 min after the start of mixing and was significantly increased at 30 min (p<0.05). There were no significant changes in compressive strength for SM from 30 to 60 min after the start of mixing (p>0.05). The compressive strength of NP significantly increased with time (p<0.05). For HG, the compressive strength significantly increased with time (p<0.05). The strength of HG at 30 min was not significantly different from that at 45 min but was significantly lower than that at 60 min. No significant change in strength was found for HG from 45 to 60 min. DS showed greater compressive strength at 30, 45 and 60 min than at 15 min (p<0.05). There were no significant changes in compressive strength values for DS from 30 to 60 min (p<0.05).
Fig. 8 Changes in linear setting expansion of dental stones used in this study with time. The reading at the time subtracted by the initial reading was divided by the initial length of specimen to obtain the linear setting expansion value at the time.

Table 9 Summary of analysis of variance for compressive strength

| Source    | df | Sum of squares | Mean square | F       | p      |
|-----------|----|----------------|-------------|---------|--------|
| Dental stone (A) | 3  | 1,408.278      | 469.426     | 86.173  | <0.001 |
| Time (B)  | 3  | 4,138.161      | 1,379.387   | 253.216 | <0.001 |
| A×B       | 9  | 1,223.613      | 135.957     | 24.958  | <0.001 |
| Error     | 64 | 348.638        | 5.447       | —       | —      |
| Total     | 79 | 7,118.690      | —           | —       | —      |

Fig. 9 Compressive strengths of dental stones used in this study.

2. Comparison of compressive strength among dental stones at each measurement time

Up to 30 min after the start of mixing, SM had the greatest compressive strength among the dental stones examined (p<0.05). No significant differences in strengths were found at 15 min between HG and DS or at 30 min between NP and HG (p>0.05). At 45 min, SM had greater compressive strength than HG and DS (p<0.05), and the strength of NP was not found to differ from those of SM or HG (p>0.05). There were no significant differences in strength between HG and DS or at 30 min between NP and HG (p>0.05). At 45 min, SM had greater compressive strength than HG and DS (p<0.05), and the strength of NP was not found to differ from those of SM or HG (p>0.05). There were no significant differences in strength between HG and DS.

Fig. 10 Relationships between density and compressive strength of dental stones at 30 and 60 min after the start of mixing. Values of “r” represent Pearson correlation coefficients.
(p>0.05) at 45 min. DS showed the lowest strength at 60 min among the stones (p<0.05). The strength of NP was greater at 60 min than that of HG (p<0.05).

3. Relationships between density and compressive strength
Figure 10 shows the relationship between density and compressive strength of dental stones at 30 and 60 min after the start of mixing. Significant positive correlation (Pearson correlation coefficient) was found between density and compressive strength at 60 min (r=0.983, p<0.05). However there was no significant correlation at 30 min (r=0.490, p>0.05).

Reproduction of details
The surface reproduction appearances are shown in Fig. 11. All dental stones examined could reproduce each marker line. Grooves a and c were observed more clearly than Groove b on each dental stone.

Marginal adaptation of the cast crown
Figure 12 shows the measured marginal discrepancies (µm, mean±SD) between the cast crowns and the master dies. The mean value of marginal discrepancy (marginal gap width) ranged from 24.2 to 29.5 µm (24.2±16.9 µm for DS, 27.1±14.0 µm for SM, 27.4±15.2 µm for HG and 29.5±10.9 µm for NP). No significant differences in marginal gap width were found among the cast crowns fabricated from the four different dental stones used (F=0.113, p=0.951, Table 10).

SEM surface observation of set dental stones
Figure 13 shows representative SEM photographs of the surface appearance of each dental stone. Needle-like precipitates supposed to be calcium sulfate dihydrate crystals appeared in all dental stones. SM revealed finer

| Source       | df  | Sum of squares | Mean square | F      | p    |
|--------------|-----|----------------|-------------|--------|------|
| Dental stone | 3   | 7.062×10^{-5}  | 2.354×10^{-5} | 0.113  | 0.951|
| Error        | 16  | 0.003          | 0.000       |        |      |
| Total        | 19  | 0.003          |             |      |      |
and denser needle-like precipitates compared with the other stones.

NP showed shorter needle-like precipitates than others. As compared to SM, thicker and larger needle-like precipitates were observed in HG. DS exhibited both needle-like and plate-like precipitates.

**DISCUSSION**

The study results indicated that, in comparison with ordinary dental stones, the newly developed SM has: 1) a much shorter setting time; 2) greater fluidity after mixing; 3) greater compressive strength at the early setting stage; and 4) less setting expansion.

SM is composed of calcium sulfate hemihydrates, as are the other stones, but the surfaces of powder particles of SM are coated with a novel surface-active agent developed by the manufacturer. This coating technology was originally developed for gypsum-bonded investment materials. For the gypsum-bonded investment materials, water reducing agents and surface-active agents are supposed to be contained in the powder, mainly for improving the handling aspects. Likewise the gypsum-bonded investment materials, some of the air entraining and high-range water reducing agents or novel surface-active agents are likely to be used to provide further fluidity for the mixture of SM. In the industrial field, chemicals such as naphthalene-based, polycarboxylic acid-based, melamine-based or aminosulfonic acid-based compound are employed as water reducing agent for the high fluidity concrete and high strength concrete to reduce the water for mixing or increase the powder surface activities. Of the water reducing agents, the polycarboxylic acid-based chemicals have been often used as a water reducing agent since 1995, because they less influence the setting time of the concrete than other agents. Although details in the additives to SM are unknown in the present study, the chemicals such as water reducing agents for concretes, surface active agents, etc are assumed to be used to manage fluidity and other properties of SM.

Since the coating treatment on the powder may increase the active surface area of stone powder particles for reaction with water, SM is considered to have a shorter setting time than the ordinary stones we examined. The higher reactivity of SM powder to water may also lead to the lower fluidity of the stone mixture. The present results showed that, although SM exhibited very high fluidity immediately after mixing, this fluidity resolved by 90 s after the start of mixing. This could be explained by the effect of the higher reactivity of SM powder to water due to the surface treatment of the powder particles or inclusion of water reducing agents.

The study results showed that SM exhibited 40 MPa of compressive strength, which satisfies the requirement for dental stone strength, by only 30 min after the start of mixing (Fig. 9). The higher reactivity of powder particles in SM would result in faster precipitation and growth of needle-like crystals of calcium sulfate dihydrate, and thus the compressive strength of stone was greater at an early period after setting.

SEM observation (Fig. 4) showed that the particle morphologies of SM were not notably different from those of the ordinary stones, but the set stones of SM revealed more tangled needle-like crystal growth compared with the ordinary stones (Fig. 13).

As shown in Table 2 and Fig. 3, smaller powder particle sizes did not always have shorter setting time or greater compressive strength in 1 h after the mixing. There are three mechanisms of gypsum setting. The most widely accepted mechanism is the dissolution-precipitation theory. Usually, finer particle have greater dissolution, and it accelerate the dissolution-precipitation process of calcium sulfate hemihydrate. Rather than the particle sizes, the chemicals such as potassium sulfate, sodium chloride or calcium sulfate dihydrate promote the crystal growth and it accelerates the setting time. It is well known that the properties of the gypsum products were depended on their purity, crystal shapes and sizes. Also the manufacturing process or additives such as retardants and accelerants is affected the precipitates formation.

This dense crystal growth in SM at the early setting stage would help to increase the compressive strength of the set stone. The dense and tangled crystal growth observed in SM would imply a reduction of spaces between the calcium sulfate dihydrate crystals, thereby reducing the setting expansion. Thus, the setting expansion of SM was less than those of the ordinary stones (Fig. 7).

The strength of set gypsum products were depended on growth of the calcium sulfate dihydrate crystal and their interlocking structure. There are unreacted calcium sulfate hemihydrate in the set materials. SM may also have some unreacted calcium sulfate hemihydrate in the set materials as seen in ordinary gypsum materials. However in this study, the amounts of unreacted calcium sulfate hemihydrate in set stones were not known and further study on effect of unreacted calcium hemihydrates on properties would be needed in future.

The porosity of gypsum products affects the dry strength, and the porosity is known to be related to water/powder ratio. The greater the water/powder ratio, the greater the porosity. As might be expected on such a basis, an increase in water/powder ratio reduces to form calcium sulfate dihydrate crystals per unit volume. Although the present study did not determine the porosity of the stones, the density of set stones was examined instead to assume the porosity, because it was supposed for the dental stones that the greater the density, the fewer the porosity. At 60 min after the start of mixing, strong positive correlation (r=0.983) was found between the density and compressive strength. The stones mixed with lower water/powder ratios exhibited greater density of set stones (Tables 1 and 6). Meanwhile at 30 min after the start of mixing, no significant correlation was observed between density and compressive strength. As seen at 60 min, the stones with lower water/powder ratios showed greater density at 30 min. However, because SM indicated much greater compressive strength at 30 min
than other stones, a significant positive correlation was not obtained in the present study. This implies that SM shows rapid increase in compressive strength during the initial 30 min after mixing as compared to other ordinary stones.

The measurement of surface detail reproduction indicated that all the dental stones examined in this study successfully reproduced the full length of the 20 µm width line without interruption (Fig. 11). SM showed good reproduction of detail, possibly due to its greater fluidity in addition to the thixotropic phenomenon produced by vibration during pouring as also seen in the ordinary stones. The results confirmed that SM offers a very similar reproducibility of detail to those of the ordinary stones mixed using a rubber bowl.

Indirect fabrication of working casts is an essential procedure for crown restorations, and the fabrication of cast crowns with good adaptability (fitting) is clinically important. The present study examined the adaptability of cast crowns to abutments using working casts prepared with SM. Following the procedure reported in previous studies, the gap widths between the crown margins and the shoulder margins of the abutments were measured. In order to clarify the difference between the dental stones used, the procedures for fabrication of cast crowns were identical for the experimental groups prepared with SM. Following the procedure reported in this study successfully reproduced the full length of the 20 µm width line without interruption (Fig. 11). SM showed good reproduction of detail, possibly due to its greater fluidity in addition to the thixotropic phenomenon produced by vibration during pouring as also seen in the ordinary stones. The results confirmed that SM offers a very similar reproducibility of detail to those of the ordinary stones mixed using a rubber bowl.

Indirect fabrication of working casts is an essential procedure for crown restorations, and the fabrication of cast crowns with good adaptability (fitting) is clinically important. The present study examined the adaptability of cast crowns to abutments using working casts prepared with SM. Following the procedure reported in previous studies, the gap widths between the crown margins and the shoulder margins of the abutments were measured. In order to clarify the difference between the dental stones used, the procedures for fabrication of cast crowns were identical for the experimental groups prepared with SM. Following the procedure reported in this study successfully reproduced the full length of the 20 µm width line without interruption (Fig. 11). SM showed good reproduction of detail, possibly due to its greater fluidity in addition to the thixotropic phenomenon produced by vibration during pouring as also seen in the ordinary stones. The results confirmed that SM offers a very similar reproducibility of detail to those of the ordinary stones mixed using a rubber bowl.

Indirect fabrication of working casts is an essential procedure for crown restorations, and the fabrication of cast crowns with good adaptability (fitting) is clinically important. The present study examined the adaptability of cast crowns to abutments using working casts prepared with SM. Following the procedure reported in previous studies, the gap widths between the crown margins and the shoulder margins of the abutments were measured. In order to clarify the difference between the dental stones used, the procedures for fabrication of cast crowns were identical for the experimental groups prepared with SM. Following the procedure reported in this study successfully reproduced the full length of the 20 µm width line without interruption (Fig. 11). SM showed good reproduction of detail, possibly due to its greater fluidity in addition to the thixotropic phenomenon produced by vibration during pouring as also seen in the ordinary stones. The results confirmed that SM offers a very similar reproducibility of detail to those of the ordinary stones mixed using a rubber bowl.

In summary, the present study suggested that the characteristics of SM are derived from the surface coating treatment of the powder particles and other ingredients such as setting controlling agents, etc. Further investigation is required to understand the effects of surface coating of powder particles on the dimensional stability of dental stones.

CONFLICT OF INTEREST

No conflict of interest exists for any of the authors of this paper. This research received no specific grant from any funding agency in the public, commercial or not-for-profit sectors.

REFERENCES

1) American dental association. Guide to dental materials and devices. sixth ed., Chicago: American dental association; 1972, p.85.
2) Shen C. Chapter 9 Gypsum products. In: Anusavice KJ, Shen C, Rawls HR, editors. Philips’ Science of dental materials,
3) Earnshaw R, Smith DC. The tensile and compressive strength of plaster and stone. Aust Dent J 1966; 11: 415-422.

4) Jørgensen KD, Kono A. Relationship between the porosity and compressive strength of dental stone. Acta Odontol Scand 1971; 29: 439-447.

5) Azer SS, Kerby RE, Knobloch LA. Effect of mixing methods on the physical properties of dental stones. J Dent 2008; 36: 736-744.

6) ISO 6873: 2013. Dentistry-Gypsum products. International Organization for Standardization, Geneva, 2013.

7) ISO 13320: 2009. Particle size analysis-laser diffraction methods. International Organization for Standardization, Geneva, 2009.

8) Kojima K, Mori D, Kumagai T. Proposal for fabrication of models by development of next generation material—Physical properties of super fast setting and fluidity dental stone. J Dent Mater 2017; 36: 354. (In Japanese)

9) PCT/JP2015/075809 WO 2016/043131 DENTAL GYPSUM—BASED EMBEDDING MATERIAL POWDER

10) Ohkawa Y. Characteristic and capacity of air entraining and high-range water reducing agents. Concrete J 1999; 37: 15-20. (In Japanese)

11) Setoyama K, Takahashi S. Crystal phase and shape control of gypsum. Inorg Mater 1995; 2: 498-505. (In Japanese)

12) Kenyon BJ, Hagge MS, Leknius C, Daniels WC, Weed ST. Dimensional accuracy of 7 die materials. J Prosthodont 2005; 14: 25-31.

13) Olivera AB, Saito T. The effect of die spacer on retention and fitting of complete cast crowns. J Prosthodont 2006; 15: 243-249.

14) Johnson R, Verrett R, Haney S, Mansuto M, Challa S. Marginal gap of milled versus cast gold restorations. J Prosthodont 2017; 26: 56-63.

15) Powers JM, Sakaguchi RL. Chapter 13 Gypsum products and Investments. In: Powers JM, Sakaguchi RL, editors. Craig’s Restorative Dental Materials. 12th ed., St. Louis: Mosby; 2006, p.324.

16) Darvell BW. Efficiency of mechanical trituration of amalgam. H. Effects of some variables. Aust Dent J 1981; 26: 25-30.