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The effects of K_2SO_4 solution on the compressive strength of dental gypsum type III

T Adeilina, S Triaminingsih*and D J Indrani

Department of Dental Materials, Faculty of Dentistry, Universitas Indonesia, Jakarta, Indonesia

*E-mail: ami_permana@yahoo.com

Abstract. Dental gypsum type III is used as a material for manufacturing working models of dentures. The aim of this study was to identify the effects of the addition of a K2SO4 solution on the compressive strength of gypsum type III. A compressive strength test was performed using a universal testing machine with a crosshead speed of 1 mm/min. The data were analyzed using a one-way ANOVA. The results showed that the compressive strength of gypsum type III with a 1.5% K2SO4 solution added was higher than for gypsum type III alone but lower than the compressive strength of gypsum type IV.

1. Introduction

Dental gypsum type III is used as a material for manufacturing working models of dentures [1]. Working models require a strong material that is not easily fractured. Dental gypsum type III contains powdered particles of calcium sulfate hemihydrate with a prismatic shape and a regular crystal [2]. The powdered particles of calcium sulfate hemihydrate in dental gypsum type IV are more regular than those in dental gypsum type III, so the ratio of water to powder in dental gypsum type IV is lower [1]. Dental gypsum type III has a minimum compressive strength of at least 20 MPa, less than that of dental gypsum type IV, which has a minimum compressive strength of at least 35 MPa [3].

K2SO4 is an effective accelerant for the hardening of gypsum. A 1.5% concentration of K2SO4 solution is the most effective because it is conveniently used by the operator [4]. A K2SO4 solution can accelerate the setting time because when mixed with powdered particles of calcium sulfate hemihydrate, it will form the compound syngenite [K2Ca (SO4)2•H2O]. Syngenite will then become the core of the calcium sulfate dihydrate crystals. Syngenite has a solubility of 2.5 g/L, which is greater than the solubility of calcium sulfate dihydrate (2.1 g/L) [5]. The higher solubility causes syngenite crystals to form faster than calcium sulfate dihydrate, so that the growth rate of calcium sulfate dihydrate crystals with a syngenite core is faster than the growth rate of calcium sulfate dihydrate crystals with a core of only calcium sulfate dihydrate. A syngenite core is larger than a calcium sulfate dihydrate core because syngenite has fewer ions, so the molecule is larger [5,6]. The larger size of the core can cause the calcium sulfate dihydrate crystals to grow larger, thus minimizing the space between them.

Dental gypsum type IV contains K2SO4 salt, which is the cause of its higher compressive strength compared to dental gypsum type III. The higher strength occurs because the space between the calcium sulfate dihydrate crystals is smaller, so the crystal structure of calcium sulfate dihydrate is denser [5]. Thus, a solution of K2SO4 is also expected to increase the compressive strength of dental gypsum type III. Dental gypsum type III with an improved compressive strength could be used as an

alternative to dental type gypsum IV. Therefore, this study measured the compressive strength of dental gypsum type III, dental gypsum type III with the addition of a 1.5% K2SO4 solution, and dental gypsum type IV.

2. Materials and Methods

In this study, the preparation of the specimens began with the amounts of water and powder specified by the manufacturer's instructions. The Group 1 specimens were made by mixing 100 g of dental gypsum type III powder with 30 ml of distilled water; the Group 2 specimens were made by mixing 100 g of dental gypsum type III powder with 30 ml of a 1.5% K2SO4 solution; and the Group 3 specimens were made by mixing 20 ml of distilled water with 100 g of dental gypsum type IV powder [4,7,8]. The dental gypsum type III that was used was produced by Moldano, and the dental gypsum type IV that was used was produced by Fujirock. The 1.5% K2SO4 solution was made by dissolving 1.5 g of K2SO4 into 100 ml of distilled water.

A test of setting time was done to ensure that a 1.5% solution of K2SO4 could work as an accelerant [4]. The test was performed using a Vicat needle and 5 specimens per group. The setting test began with the production of the specimens in each of the groups. The dental gypsum dough was manipulated using a vacuum gypsum mixer. The dough was then cast into a 50-ml pot and placed under the Vicat needle. The Vicat needle was penetrated with an interval of 15 seconds. The setting time test was considered to be finished when the Vicat needle could not penetrate >2 mm into the dental gypsum dough [3].

The compressive strength test was performed using a universal testing machine (UTM). There were 7 specimens in each group. The three groups of dental gypsum specimens were made by mixing water and gypsum powder according to the manufacturer's instructions using a vacuum gypsum mixer, and the dental gypsum was cast into a stainless steel mold with a diameter of 20 mm and a height of 40 mm. The UTM had a load of 2,500 kgf and a test speed of 1 mm/min. The tests were performed at 1 hour, 24 hours, and 7 days after the production of the specimens.

3. Results and Discussion

3.1 Results

Setting time tests were conducted on Group 1 (dental gypsum type III), Group 2 (dental gypsum type III with a 1.5% K2SO4 solution added), and Group 3 (dental gypsum type IV) specimens. The average setting time are listed in Table 1. The setting time test results showed a difference between the setting timeof dental gypsum type III with and without 1.5% K2SO4; the use of K2SO4 reduced the setting timeof dental gypsum type III. As seen in Table 1, dental gypsum type III without K2SO4 had a setting timeof 10 minutes, 36 seconds; with K2SO4, the setting timewas 4 minutes, 56 seconds. Dental gypsum type IV had a setting timeof 10 minutes, 3 seconds. The values obtained from the compressive strength tests can be seen in Table 2.

Table 1.Setting time of dental gypsum type III, dental gypsum type III with a 1.5% K2SO4 solution, and dental gypsum type IV

Average Setting Time	
Group	**Setting Time ± SD**
(1) Dental gypsum type III	10 minutes, 36 seconds ± 18 seconds
(2) Dental gypsum type III with a 1.5% K2SO4 solution	4 minutes, 56 seconds ± 7 seconds
(3) Dental gypsum type IV	10 minutes, 3 seconds ± 9 seconds

Table 2. Compressive strength of dental gypsum in the three specimen groups

Specimen groups	Compressive strength (MPa) ±SD		
	1 hour	24 hours	7 days
(1) Dental gypsum type III + distilled water	20.33 ± 0.39	22.01 ±0.36	29.05 ±1.06
(2) Dental gypsum type III + 1.5%K_2SO_4 solution	21.96 ±0.33	23.31 ±0.35	23.72 ±0.49
(3) Dental gypsum type IV + distilled water	37.94 ±1.28	44.82 ±1.3	44.85 ±1.3

A Shapiro-Wilk test showed that the compressive strength test data for Group 1 at 1 hour, 24 hours, and 7 days had a normal distribution. A test of the Levene statistic showed that the data were not homogeneous, so a Welch correction was made so that the data could be tested with a one-way ANOVA. The comparison of the average compressive strengths in Group 1 at the three different time $p < 0.05$. This shows that there was a significant difference among the groups. A Games-Howell post-hoc test was performed to compare the compressive strengths in a pairwise fashion. In Group 1, there was a significant difference between the average compressivestrengths of the 1-hour and 24-hour specimens ($p = 0.020$). There was also a significant difference between the average strengths of the 1-hour and 7-day specimens ($p = 0.00$) and between the 24-hour and 7-day specimens ($p = 0.00$). A Shapiro-Wilk normality test of the compressive strength data for Group 2 showed a normal distribution. A test of the Levene statistic showed that Group 2 had homogeneous data. A one-way ANOVA test showed a significant difference in the average compressive strengths among the three different time points ($p = 0.041$). A post-hoc Bonferroni test was conducted to examine the results in more detail. For Group 2, there was no significant difference between the average compressive strengths of the 1-hour and 24-hour specimens ($p = 0.181$), between the 1-hour and 7-day specimens ($p = 0.052$), or between the 24-hour and 7-day specimens ($p > 0.05$).

A Shapiro-Wilk test showed that the compressive strength data for Group 3 had a normal distribution. A test of the Levene statistic showed that the strength data were homogeneous. A one-way ANOVA was performed to test for strength differences among the three time points; a significant difference was found ($p = 0.002$). A post-hoc Bonferroni test was performed to examine the results more closely. For Group 3, there was a significant difference between the average compressive strengths of the 1-hour and 24-hour specimens ($p = 0.005$), between the 1-hour and 7-day specimens ($p = 0.005$), and between the 24-hour specimens and the 7-day specimens ($p < 0.05$). Another set of comparisons was made among the 1-hour specimens from Groups 1, 2, and 3. A Shapiro-Wilk test showed a normal distribution of the data. However, a test of the Levene statistic showed that the data distribution was not homogeneous. A Welch correction was used to correct the data so that they could be tested with a one-way ANOVA, which showed a significant difference among the compressive strengths of the three groups ($p = 0.00$). A post-hoc Games-Howell test showed a significant difference in the compressive strengths of the 1-hour specimens between Groups 1 and 2 ($p = 0.024$), between Groups 1 and 3 ($p = 0.00$), and between Groups 2 and 3 ($p = 0.00$).

The data for the 24-hour specimens from Groups 1, 2, and 3 had a normal distribution. However, the Levene test statistic showed that the data were not homogeneous, so a Welch correction was performed so that the data could be tested with a one-way ANOVA. There was a significant difference among the average strengths of the 24-hour specimens in Groups 1, 2, and 3 ($p = 0.00$). The results of a post-hoc Games-Howell test showed that there was no significant difference between the average compressive strengths in Groups 1 and 2 ($p = 0.175$). However, there was a significant difference between the average compressive strengths in Groups 1 and 3 and between Groups 2 and 3 ($p = 0.00$ for both). A Shapiro-Wilk test showed that the data from the 7-day specimens in Groups 1, 2, and 3 had a normal distribution. A test of the Levene statistic showed that the data were not homogeneous, so a Welch correction was performed so that the data could be analyzed with a one-way ANOVA. There was a significant difference among the average compressive strengths in Groups 1, 2, and 3 ($p =$

0.00). A post-hoc Games-Howell test was performed to examine the results more closely. There was a significant difference between the average compressive strengths of the 7-day specimens for Groups 1 and 2 ($p = 0.014$), for Groups 1 and 3 ($p = 0.00$), and for Groups 2 and 3 ($p = 0.00$).

3.2 Discussion

To test the possibility of accelerating the setting time of dental gypsum type III, it was manipulated by the addition of a 1.5% K2SO4 solution. The setting time of dental gypsum type III manipulated with only distilled water was 10 minutes, 36 seconds, while the setting time of dental gypsum type III manipulated with the addition of a 1.5% K2SO4 solution was 4 minutes, 56 seconds. This is because the use of a K2SO4 solution causes the formation of the compound K2SO4H2O, or syngenite, which then forms the core of the calcium sulfate dihydrate crystals. Syngenite has a high solubility (2.5 g/L) compared to calcium sulfate dihydrate (2.1 g/L). The crystal growth rate of calcium sulfate dihydrate with a syngenite core is faster than the crystal growth rate of calcium sulfate dihydrate with a core of calcium sulfate dihydrate, because a nucleus of syngenite is formed faster. This core is the origin of the formation of the arms of the calcium sulfate dihydrate crystals. The arms will grow in all directions, following the form of the core, so that there will be contact between the crystals; the contacts accelerate the hardening of the dental gypsum [5]. The results of the setting time tests in this study accord with previous research concerning the influence of various concentrations of K2SO4 salt on the hardening of dental gypsum type III. The research states that the setting time of dental gypsum type III manipulated with a 1.5% solution of K2SO4 is the most effective because it is the easiest for the operator to handle [4].

Tests of the compressive strength of dental gypsum type III made with distilled water were performed at 1 hour, 24 hours, and 7 days after the production of the samples. The results showed an improvement in compressive strength over time. The test of the wet strength of gypsum type III, which took place 1 hour after the specimen production, showed a compressive strength of 20.33 MPa. At this stage, the calcium sulfate dihydrate crystals are forming with a nucleus of calcium sulfate dihydrate. The test of dry strength, 24 hours after the specimens were made, showed an increase in the value of the compressive strength to 22.01 MPa. This is because during the storage of the dental gypsum specimens, the water between the crystals evaporates, causing a calcium sulfate dihydrate residue to form. This residue is deposited between the calcium sulfate dihydrate crystals, causing the bonds to become stronger. Therefore, there is an improvement in the compressive strength. After 7 days, the compressive strength increased to 29.05 MPa. This is because when dental gypsum is left to dry in the open air, which has a moisture content of 55%, evaporation still occurs, causing more calcium sulfate dihydrate residue to affix to the calcium sulfate dihydrate crystals [5]. The more excess water that evaporates, the more the compressive strength increases.

The compressive strength of dental gypsum type III with an added solution of 1.5% K2SO4 was tested at 1 hour, 24 hours, and 7 days after the production of the specimens. At 1 hour, the compressive strength was 21.96 MPa. At this stage, the K2SO4 solution causes the formation of syngenite, which becomes the core of the calcium sulfate dihydrate crystals. Syngenite has a higher solubility than calcium sulfate dihydrate, so the syngenite nucleus is more quickly formed. The faster core formation causes more calcium sulfate dihydrate crystals to form so that the the crystal structure becomes denser. In addition, the syngenite nucleus has a large size because it contains many syngenite molecules. A larger core causes the arms of the calcium sulfate dihydrate crystals to become larger and minimize the space between the calcium sulfate dihydrate crystals. In testing the dry strength 24 hours after the specimens were made, the compressive strength was found to be 23.31 MPa. This increase in the value of the compressive strength is not significant because the space between the calcium sulfate dihydrate crystals is small. The compressive strength of the 7-day specimens (23.72 MPa) was not significantly higher than that of the others. This is expected, because the water in the calcium sulfate dihydrate has evaporated at 24 hours after the specimen production. At 7 days, there is no more water to be evaporated, so the strength tends to remain constant.

At 1 hour after manipulation, the compressive strength of the dental gypsum type III made with K2SO4 was significantly higher than the compressive strength of the dental gypsum type III made with distilled water. This is due to the formation of syngenite in the gypsum with K2SO4. Syngenite can accelerate the growth rate of calcium sulfate dihydrate crystals. In the dry-strength (24-hour) test, the strength of gypsum type III with K2SO4 was not significantly higher than that of the gypsum type III made with distilled water. This is because the space between the calcium sulfate dihydrate crystals is smaller in the gypsum made with K2SO4. Thus, there is less residual calcium sulfate dihydrate in between the calcium sulfate dihydrate crystals, so the process of evaporation of the calcium sulfate dihydrate solution does not have a great impact on the compressive strength. In addition, for the 7-day specimens, the strength of the dental gypsum type III manipulated with K2SO4 was lower than the strength of the dental gypsum type III made with distilled water. This is expected that when gypsum is made with K2SO4, the water evaporates within 24 hours, whereas in regular gypsum, some water still remains on day 7.

The compressive strength of the dental gypsum type IV at 1 hour was 37.94 MPa. This is because dental gypsum type IV has a high concentration of K2SO4 (4%) [4]. A high concentration of K2SO4 causes a higher number of syngenite cores to form, so that the crystal structure of the calcium sulfate dihydrate will be denser. The compressive strength of gypsum type IV at 24 hours (44.82 MPa) was significantly greater than the strength at 1 hour. A significant increase is expected because the concentration of K2SO4 is high, which causes the syngenite cores to continue to grow after the 1-hour test. This is because the syngenite nuclei will continue to grow until some of the K+ ions are used in the formation of calcium sulfate dihydrate crystals [6]. However, the compressive strength of the dental gypsum type IV at 7 days (44.85 MPa) was not significantly higher than the others. At this stage, the high concentration of K2SO4 causes there to be less space between the calcium sulfate dihydrate crystals so that there is less residual calcium sulfate dihydrate solution between the crystals. With little solution present, the process of evaporation does not significantly increase the compressive strength of the dental gypsum type IV 7 days after it was made.

Dental gypsum type IV had a higher compressive strength than the dental gypsum type III made with K2SO4. This is due to the high concentration of K2SO4 in dental gypsum type IV (4%) which is added by the factory to reduce the expansion of dental gypsum. The high concentration of K2SO4 in gypsum type IV also causes more syngenite to form [9], leading to a greater number of syngenite cores, a higher production of calcium sulfate dihydrate crystals, and a reduction in the space between the calcium sulfate dihydrate crystals. Based on this study, the use of a 1.5% K2SO4 solution can increase the compressive strength of dental gypsum type III at 1 hour after manipulation. However, the compressive strength of dental gypsum type III manipulation with K2SO4 is not as high as that of dental gypsum type IV. This is expected, because a concentration of 1.5% is not able to significantly improve the crystal density of calcium sulfate dihydrate. Therefore, due to its lower compressive strength, dental gypsum type III made with 1.5% K2SO4 cannot functionally replace dental gypsum type IV.

4. Conclusion

A 1.5% K2SO4 solution improves the compressive strength of dental gypsum type III on 1 hour after manipulation. However, dental gypsum type III manipulation with 1.5% K2SO4 cannot be an alternative to dental gypsum type IV, when viewed from the compressive strength.

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