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The effect of K_2SO_4 solution on type III gypsum surface roughness

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Abstract. The working model of type III gypsum is commonly used as working model for removable dentures. K_2SO_4 is well known as an effective accelerator to accelerate the gypsum setting time. This study aimed to identify the effect of K_2SO_4 1.5% solution on type III gypsum surface roughness. Surface roughness tests were performed using a Surface Roughness Tester at 1 hour, 24 hours, and 7 days after manipulation the gypsum. The results showed that type III gypsum surface roughness varied until the 7-day test. Moreover, the surface roughness of type III gypsum and K2SO4 1.5% solution is lower than type III gypsum surface roughness and equal to type IV gypsum surface roughness. It is concluded that the addition of K2SO4 1.5% solution decreased type III gypsum surface roughness.

1. Introduction

Gypsum material (CaSO4.2H2O) is widely used in dentistry as an impression, model, cast, and die material. Gypsum material is classified into 5 types: type I impression plaster, type II model plaster, type III dental stone, type IV dental stone high strength low expansion, and type V dental stone high strength high expansion [1]. Every type of gypsum has its own function. Type 1 gypsum is used as an impression material; type II gypsum is used in flasking processes and for study models; type III gypsum is used for working models; and type IV and type V are used as die materials [2].

The working model of type III gypsum is commonly used in the manufacture removable dentures because of its high strength, good surface reproducibility, and affordability compared to other gypsum products [2]. Moreover, type III gypsum has low surface roughness. The roughness score of type III gypsum is around 0.45 μm [3]. However, the surface roughness value may be affected by prolong contact with alginate. Prolong contact between gypsum and alginate in the setting state can make the model surface fragile, easily abraded, and rough because of the water content in alginates [4,5]. Thus, to prevent the problem of contact duration with alginate, the accelerator is added to reduce the gypsum setting time [4]. K2SO4 is well known as an effective accelerator to accelerate the gypsum setting time [2]. The acceleration of gypsum setting time is due to the addition of K2SO4, which will later form the syngenite [$K_2Ca(SO_4)_2$. H_2O] during the crystallization process [6]. Syngenite is the result of K2SO4 reaction with gypsum ($CaSO_{4.2}H_2O$), which will become the core of gypsum crystal growth and replace sulfate dihydrate. Syngenite has a higher solute than calcium sulfate dihydrate, so the gypsum crystal growth core will form quickly and the crystal will accelerate growth.

Besides accelerate the gypsum setting time, the use of K_2SO_4 1.5% solution can change the gypsum crystal arrangement to become denser and more regular [7]. This is due to the high solubility, and also crystal syngenite larger than calcium sulfate dihydrate. The denser gypsum structure is predicted to affect the gypsum properties, including the surface. The dense gypsum surface structure is expected to refine the gypsum structure. Thus, the predicted K_2SO4 solution not only accelerate the setting time, but also decreases the roughness of the Type III gypsum surface. The concentration of K2SO4 solution should be carefully calculated since not all concentrations in gypsum manipulation will give optimal results.

Conversely, type IV gypsum has a lower surface roughness than the coarse surface roughness of type III gypsum of about 0.37 μm [8]. The smooth surface of type IV gypsum is thus one of the criteria for making die for indirect restoration. However, type IV gypsum is more expensive than type III gypsum. The use of a 1.5% K2SO4 solution in type III gypsum is expected to create a smoother surface as an alternative to type IV gypsum.

2. Materials and Methods

The gypsum setting time test is performed as a preliminary experiment, and the surface roughness test is performed on three groups: type III gypsum, type III gypsum with K2SO4 1.5% solution, and type IV gypsum. The specimen is created through the manipulation of gypsum with the ratio of water and powder following the manufacture procedure, which is 30 ml of aquades and 100 g of gypsum powder for the type III gypsum group; 30 ml of K2SO4 1.5% solution and100 g of gypsum powder for the type III gypsum group; and 20 ml of aquades and 100 g of gypsum powder for the type IV gypsum group. The type III gypsum is from Moldano, and the type IV gypsum is from Fujirock. The K2SO4 1.5% solution is made by adding 1.5 g of K2SO4 salt to 100 ml of aquades.

The setting time test is used to confirm that the K2SO4 solution can accelerate the type III gypsum. Five specimens are needed for each group, and a gypsum mixer is used to manipulate the gypsum. The gypsum dough is poured into a 50-ml plastic jar. Then, the test is conducted using a vicat needle. One or two minutes before the expected setting time, the vicat needle is released to penetrate the dough every 15±1 second. The setting time is counted from the start of mixing until the vicat needle penetrates the specimen at a 2-mm depth.

The surface roughness test is conducted at three different times: 1 hour, 24 hours, and 7 days after manipulation. Seven specimens are needed for each group. The specimen measured 1 hour after manipulation are stored in the open jar at room temperature with a mean humidity around 55% until the next measurements at 24 hours and 7 days after manipulation. Each specimen's surface roughness is measured with a Surface Roughness Tester (Mitutoyo SJ-301) and using the parameter of surface roughness Ra to show the mean surface roughness. The cut of length used is 0.8 mm (n =5), and the evaluation length is 4 mm. Every specimen is tested five times at different random places to get the mean surface roughness from one specimen.

3. Results and Discussion

3.1 Results

The result of the time setting test is used as a guideline in gypsum manipulation for the preparation of surface roughness specimens for gypsum type III, type III gypsum with 1.5% K_2SO_4 solution, and IV type gypsum at 1 hour (wet strength), 24 hours (dry strength), and 7 days after manipulation. The mean value of setting times of type III gypsum, type III gypsum with K_2SO_4 1.5% solution, and type IV gypsum are shown in Table 1

Table 1. Mean value of gypsum setting time

Specimen group	Setting time± SD (mins)
Type III gypsum	10 mins and 36 seconds ± 18 seconds 10.60 ± 0.30
Type III gypsum + K^2SO^4 1.5% solution	4 minutes and 56 seconds ± 7 seconds 4.933 ± 0.11
Type IV gypsum	10 minutes and 3 seconds ± 9 seconds 10.05 ± 0.15

Table 1 shows that the type III gypsum and K_2SO_4 1.5% solution has faster setting time compared to the type III and type IV gypsum groups. The setting time of the K_2SO_4 1.5% solutions is 4 minutes and 56 seconds. This setting time is approximately 200% faster than the type III gypsum group time (10 minutes and 36 seconds) and the type IV gypsum group time (10 minutes and 3 seconds).

In the preliminary experiment, it is proven that the K_2SO_4 1.5% solution effectively accelerates the gypsum setting time. Next, the surface roughness test is conducted to identify the effect of the surface roughness. The mean value of type III gypsum, type III gypsum with K_2SO_4 1.5% solution, and type IV gypsum measured at 1 hour, 24 hours, and 7 days after manipulation are shown in Table 2.

Table 2. The mean value of surface roughness test, Ra (μm)

Gypsum	Surface roughness at different measuring time Mean ± SD (μm)			Sig*
	1 hour	24 hours	7 days	
Type III	1.52 ± 0.07	1.57 ± 0.12	1.32 ± 0.07	0.000*
Type III + K_2SO_4 1.5% solution	0.82 ± 0.07	0.85 ± 0.06	0.79 ± 0.08	0.237
Type IV	0.90 ± 0.08	0.83 ± 0.06	0.84 ± 0.09	0.235
Sig*	0.000*	0.000*	0.000*	

*$p < 0.05$: statistically significant, one-way ANOVA test

Table 2 shows the mean value of all gypsum types measured at 1 hour, 24 hours, and 7 days after manipulation. The results of the statistical test using a one-way ANOVA show that the mean surface roughness value from type III gypsum at measurement times of 1 hour, 24 hours, and 7 days after manipulation is 0.000 ($p < 0.05$). This means that the surface roughness mean value of type III gypsum is statistically significant.

However, the mean value of the surface roughness test in the type III gypsum with K_2SO_4 1.5% solution group and in the type IV gypsum group at 1 hour, 24 hours, and 7 days after manipulation are 0.237 ($p > 0.05$) and 0.235 ($p > 0.05$), respectively. This means that the mean value of the surface roughness test in these two groups is statistically insignificant. After the post hoc Bonferroni statistic test on the type III gypsum group, the mean value of the surface roughness test 1 hour after manipulation compared to 24 hours after measurement is found to be statistically insignificant ($p > 0.05$). The mean value of the surface roughness test at 1hour measurements compared to 7daymeasurementswasstatisticallysignificant ($p < 0.05$). Also, the mean surface roughness test values at 24 h and 7 days after manipulation showed a statistically significant decrease ($p <0.05$).

The result of the statistical test using a one-way ANOVA show that the significance mean surface roughness value between the three groups of gypsum at1 hour, 24 hours, and 7 days after

manipulation are 0.000 ($p < 0.05$). This means that the mean surface roughness test on type III gypsum, type III gypsum with K_2SO_4 1.5% solution, and type IV gypsum at each measurement are statistically significant. The post hoc Bonferroni was tested to determine the significance of surface roughness value between each group at each measurement time. The results of each measurement time are the same; the mean value of the type III gypsum group and the type III gypsum with K_2SO_4 1.5% solution group presented a statistically significant difference ($p < 0.05$). Next, the mean surface roughness value between the type III gypsum and type IV gypsum groups also present a statistically significant decrease ($p < 0.05$). The mean surface roughness test between type III gypsum with K2SO4 1.5% solution and type IV gypsum groups do not present a statistically significant decrease ($p > 0.05$). The mean surface roughness of all groups at1 hour, 24 hours, and 7 days after manipulation are shown in Figure1.

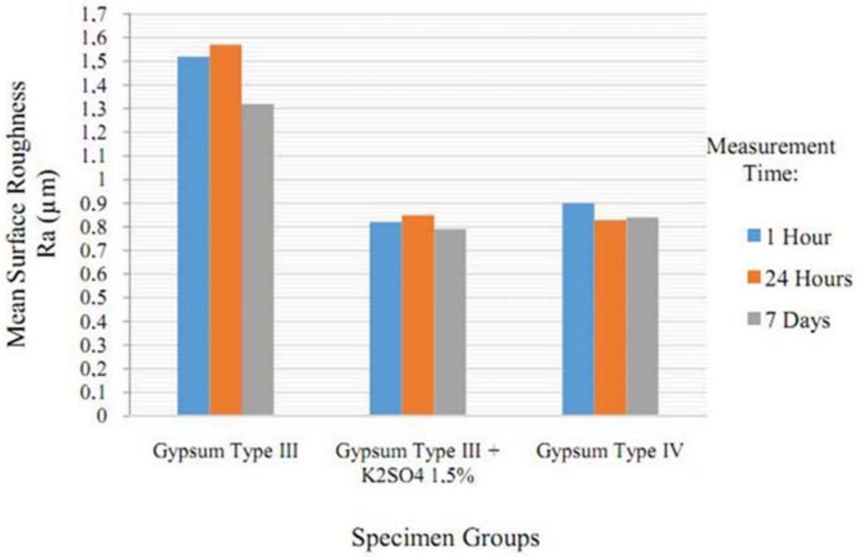

Figure1. Mean surface roughness test value, Ra (µm)

3.2 Discussion

On this research, the preliminary experiment that is the setting time test was done as a guideline for preparation the surface roughness tests specimen of type III gypsum, type III gypsum with K_2SO_4 1.5% solution, and type IV gypsum. The results of the preliminary experiment prove that a K_2SO_4 1.5% solution effectively accelerates the setting time of type III gypsum. This is proven by the difference between the setting time of type III gypsum without a K_2SO_4 solution (10 minutes and 36 seconds) compared to the setting time of type III gypsum with K2SO4 1.5% solution (4 minutes and 56 seconds). The experiment results match to the other experiment results regarding the effect of K_2SO_4 salt concentration on the setting time of type III gypsum and also proved that K_2SO_4 can accelerate gypsum setting time without affecting the operator to manipulate the gypsum [9]. This is because the calcium sulphate hemihydrate that reacts with the K_2SO_4 1.5% solution later releases Ca2+ and SO42-, K+ and SO42-, and H+ and O2- ions. These ions bind to each other and make a sediment that creates a mixed compound [K2Ca (SO4)2.H2O] known as syngenite [6]. Syngenite also creates the spherulite formation and becomes the growth core of calcium sulphate dihydrate. The presence of syngenite, which replaces the calcium sulphate dihydrate as a growth core, differentiates the reaction of gypsum setting times between groups with and without K_2SO4. Syngenite can bind two SO42- ions to one Ca2+ ion that has higher solubility (2.5 g/l) compared to calcium sulphate dihydrate solubility (2.1 g/l). This makes the syngenite faster to form and will later enhance the growth of calcium sulfate dihydrate in one solution at the same time. Thus, the crystal growth spurt is elevated and the spherulite crystals form and bind with each other faster. This accelerates the gypsum setting time [10].

After measuring the setting time in the preliminary experiment, surface roughness tests on type III gypsum, type III gypsum with K_2SO_4 1.5% solution, and type IV gypsum were conducted at 1 hour, 24 hours, and 7 days after manipulation. The mean surface roughness value on the measurement at 1hour and 24 hour were statistically insignificant, but surface roughness value presented significant decreases between 24 hour and 7 day measurements after manipulation. This is called the ageing process, and significant increase in the strength of hardened gypsum can be seen 24 hours after manipulation. The ageing process caused by the evaporation of calcium sulphate dihydrate from the hardened gypsum. The evaporation process leaves residuals from the calcium sulphate dihydrate solution and other substances that fill the spaces between calcium sulphate dihydrate crystals and binds them together. This causes the space between gypsum crystals reduce and decrease the gypsum surface roughness. Therefore, the lowest surface roughness value was obtained at 7 days after manipulation from the type III gypsum with K2SO4 1.5% solution group [10].

The results of the type III gypsum with K2SO4 1.5% solution group were not statistically different at1hour, 24 hour, and 7 day measurements after manipulation. In other words, they were essentially constant over time. This was due to the use of the K_2SO_4 1.5% solution, which accelerates the arrangement of denser calcium sulphate dihydrate crystals compared to gypsum groups without the K2SO4 solution. In the latter groups, the space between calcium sulfate crystals was also smaller. Hence, the evaporation process as measured 7 days after manipulation did not have a significant effect on decreasing the surface roughness of the type III gypsum with K_2SO_4 1.5% solution. The dense arrangement of quickly formed calcium sulphate dihydrate crystals is the result of two mechanisms [4,10]. The first mechanism is caused by the increasing of calcium sulphate dihydrate crystals as a growth core, which will later form as syngenite. Syngenite as a growth core of calcium sulphate dihydrate crystals is more formed at the 1 hour than calcium sulphate dihydrate as a growth core of another crystal from type III gypsum without the K2SO4 solution [7]. More syngenite more quickly increases the amount of calcium sulphate dihydrate formed and makes the gypsum structure denser. The second mechanism is caused by the bigger syngenite crystal compared to the calcium sulphate dihydrate as a growth core of another crystal. This is because the syngenite has a molecule addition from the K2SO4 solution [7,10]. According to studies on the effect of the K2SO4 solution on gypsum crystallization with Scanning Electron Microscope, calcium sulphate dihydrate from syngenite is bigger than crystals from the calcium sulphate dihydrate core [7]. The bigger crystal decreases the space between crystals and makes the arrangement of calcium sulphate dihydrate denser. As a result, the evaporation process at the 24hour and 7day do not significantly impact a decrease in surface roughness.

Surface roughness value of type IV gypsum measured at 1 hour, 24 hours, and 7 days after manipulation also do not have a significant difference. This is because the type IV gypsum powder is denser and has a more regular shape. Hence, the differences between water and powder type IV gypsum are minimal (0.20) [2]. This arrangement increases the calcium sulphate dihydrate density and decreases the space between crystals compared to type III gypsum [10]. Other than that, the content of K2SO4 inside the type IV gypsum composition is 4% [10,11]. This affects the syngenite formed during the type IV gypsum crystallization as well as the denser arrangement of the calcium sulphate dihydrate crystal. As a result, the evaporation process matches that of the type III gypsum with K2SO4 1.5% solution group: from the 1hour to the 7 days, the type IV gypsum did not have a significant effect because there was little space left that the calcium sulphate dihydrate residue could fill following the evaporation process. The density change that causes a difference in the surface roughness of type IV gypsum thus does not significantly change or tends to be constant because the density is compact from the beginning.

The surface roughness test results show that the significant value decrease presents in type III gypsum and type III gypsum with K2SO4 1.5% solution groups at every measurement. This demonstrates that the K2SO4 1.5% solution can be used to decrease the surface roughness of type III gypsum. This is caused by two mechanisms of the K2SO4 1.5% solution that increase the density of the calcium sulphate dihydrate crystal arrangement as previously explained so that the syngenite core that is formed in the solution combined with the large size of the syngenite decreases the space between crystals. The denser arrangement of calcium sulphate dihydrate crystals makes the surface roughness of type III gypsum and K2SO4 1.5% solution smoother [6,7,10].

Gypsum surface roughness type III value with 1.5% K2SO4 solution according to gypsum type IV at all measurements. The gypsum type IV has a finer surface roughness because the water and powder ratios are smaller and because of the K2SO4 content contained therein, the calcium sulfate crystals crystallate becomes denser [6]. The content of K2SO4 in type IV gypsum also influences the mechanism of syngenite in space on crystalline calcium sulfate dihydrate [7,10,11]. After completing the surface roughness test, it can be concluded that 1.5% K2SO4 added to type III gypsum produces the same roughness with surface roughness in IV type gypsum and subtler than roughness in Type III gypsum without K2SO4 solution.

4. Conclusion

K_2SO_4 can be used to decrease the surface roughness value of type III gypsum surface and resembles the surface roughness value of type IV gypsum. Although K2SO4 may be used to decrease gypsum surface roughness, its clinical effectiveness is still unknown because gypsum is in direct contact with alginates.

References

[1] American National Standars/American Dental Association Specification. No 25 2000 *Dental gypsum product*s. (USA: Standard N. ANSI/ADA) p. 244-53.

[2] Rawls H R, Shen C and Anusavice K J 2013 *Phillip'S Science of Dental Materials. 11th ed. J.Chem. Inform. Model. 53*(St. Louis: ELSEVIER) p. 1689-99.

[3] Cilingir A, Geckili O, Parlar Z, Gencel B, Bozdag E and Temiz V 2013 The influence of steam cleaning procedures on the surface roughness of commonly used type III dental stone for the fabrication of removable dentures. *Gerodontol. J.***30** 171–5.

[4] Sakaguchi R L and Powers J M 2012 Craig's Restorative Dental Materials. 13th ed. (Philadephia: ELSEVIER) p. 277.

[5] Marquezan M, Jurach E M, Guimarães V D, Valentim R G A, Nojima L I, Nojima M D C G 2012 Does the contact time of alginate with plaster cast influence its properties? *Braz. Oral.Res.* 26197–201.

[6] Phillips R W1973 *Skinner's Science of Dental Materials*. 7th ed. (Philadelphia: W.B. Saunders Co.) p. 55-82.

[7] Shen C and Kamar A 1981 Effect of K2S04 and CaSO4 Dihydrate solutions on crystallization and strength of gypsum. *J. Dent.* **60** 1410–8.

[8] Cesero L De, Mota E G, Burnett L H, Spohr A M and Alegre P 2014 The influence of postpouring time on the roughness, compressive strength, and diametric tensile strength of dental stone. *J. Prosthet. Dent*. **112** 1573–7.

[9] Wardhany I 1998 *The effect of various concentration of K2SO4 salt on setting time of dental stone type III (Moldano) and type IV (Glastone).* BachelorTheses (Jakarta: Fakultas Kedokteran Gigi Universitas Indonesia).

[10] Darvell B W 2009 *Materials Science for Dentistry*. 9th Ed. (Cornwall, UK: Woodhead Publishing Limited) p. 37-59

[11] Hcs O 2015 *Safety Data Sheet. Mater Saf Data Sheet GC FUJIROCK.0* (USA: GC America Inc.) p. 2–1.