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Original Paper

Setting properties and sealing ability of hydraulic temporary sealing materials

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This study sought to investigate the setting progress and sealing ability of hydraulic temporary sealing materials used in endodontic treatment: Lumicon, Caviton, and HY-Seal. To evaluate setting progress, the materials were filled into glass tubes with one end sealed and immersed in water. After immersion, a measurement apparatus was inserted from the non-immersed end and the set area was determined by subtracting the unset area from the sample thickness. To evaluate sealing ability, materials were filled into glass tubes and divided into four groups based on different immersion times. Thermal cycling and dye penetration were performed. At 7 days, the setting depths of HY-Seal and Caviton were almost equivalent to full sample thickness, while that of Lumicon was only half of full sample thickness ($p<0.01$). On sealing ability, Lumicon ranked the highest followed by Caviton, whereas HY-Seal was unstable ($p<0.01$). These results suggested that there was no correlation between setting progress and sealing ability.

Key words: Sealing ability, Setting, Temporary filling materials

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INTRODUCTION

To seal a root canal temporarily, good filling materials play a pivotal role in keeping the root canals sterile. In today's endodontic treatment, hydraulic temporary sealing materials, zinc oxide-eugenol cements, and resin-based cements are commonly used as temporary filling materials. In particular, hydraulic temporary sealing materials are putty materials which are easy to use because they do not require mixing. The desired amount can be taken out of a container and inserted into a cavity for sealing.

Hydraulic temporary sealing materials are based on calcium sulfate: they set upon contact with saliva in the oral cavity. During setting, the materials begin to chemically react and adhere to dentin as they undergo linear hygroscopic expansion like plaster, thereby resulting in good sealing ability[1-5]. However, as water gradually permeates into the material, the setting time will be extended. Some manufacturers have indicated in their manuals that the complete setting time is 30 minutes, but other manufacturers direct dentists and patients to avoid mastication or any mechanical stress for one hour. Thus, the setting time varies greatly among materials. It is noteworthy that in general, a prolonged setting time decreases the mechanical strength of temporary filling materials[6].

The setting reaction of hydraulic temporary sealing materials is different from other dental cements. As such, no ISO standard tests can be applied to assess the setting properties of these materials. Coupled with scarce reviews for setting as compared to sealing, their setting characteristics remain unclear to date. Against this backdrop of information scarcity, this study was conducted to investigate the setting progress of hydraulic temporary sealing materials and its relationship with sealing.

MATERIALS AND METHODS

Experiment 1 — Setting test

1. Sample preparation

Table 1 lists the temporary filling materials used in this study. The materials were filled into glass tubes of 8 mm diameter and 5 mm depth. To simulate dentin, the inner surface was roughened with a carborundum stone, and then washed and dried[7]. For only one side of the sample to contact water during immersion, one end of each glass tube was sealed with paraffin wax. Then, using a plastic instrument, each temporary filling material was filled into the glass tubes sealed at one end. The surface was made flat using a cotton pellet moistened with distilled water. The samples were then immersed in distilled water and stored in an incubator at 37°C (SLV-11, Isuzu Seisakusho Co. Ltd., Tokyo, Japan). To examine the influence of immersion every day, three samples of each material were prepared for each day. One immersion cycle consisted of a period from 1 to 7 days. Five cycles were repeated.

Table 1 Temporary filling materials used in this study

Material	Composition*	Lot. No.	Manufacturer
Caviton	Zinc oxide, Plaster of Paris, vinyl acetate, others	107171	GC Corporation Tokyo, Japan
Lumicon	Plaster, Zine Oxide, Zinc sulfate	150188	Heraeus Kulzer Japan Co., Ltd. Tokyo, Japan
Hy-Seal	Dental Stone, Glycerin acetic ester, Polyvinyl choloride/acetic vinyl copolymer, Calcium sulfate, Titanium dioxide, Tannic acid, others	70123	SHOFU INC. Kyoto, Japan

*As reported by Manufacturer.

2. Measurement of setting depth

After samples were removed from water immersion, the paraffin wax was also removed to expose the side of the samples not immersed in water. The thickness of each sample was measured with digital calipers (Digimatic Caliper No. 500-120, Mitsutoyo, Tokyo, Japan) as D_1. At the non-immersed side, a measurement needle was inserted and the depth of the unset area was measured using a texture analyzer (EZ Test, Shimadzu Corp. Ltd., Kyoto, Japan).

After the sample was secured on an exclusive mold with the non-immersed side facing up, the measurement needle of about 1 mm diameter with a flat end was lowered from the non-immersed side toward the set area at a speed of 1 mm/s. As the measurement needle was lowered, the load it sensed was recorded using a data processing software (Trapezium, Shimadzu Corp. Ltd., Kyoto, Japan). The measurement needle was assumed to have reached the set area when the load limit was reached — which was predetermined at 2 kgf. In the meanwhile, the penetration depth was defined as the depth of the unset area, D_2 (Fig. 1). When the load reached 2 kgf during penetration, it was regarded as the maximum load point. Setting depth X was then calculated from the difference between D_2 and D_1 (as shown below). Since measurement needle penetration destroyed the sample, each sample was measured only once.

$$D_1 \text{ (Sample thickness)} - D_2 \text{ (Depth of unset area)} = X \text{ (Setting depth)}$$

3. Statistical analysis

Based on the mean values, temporal changes of the setting depth in each cycle were expressed as a line graph. Using an image analysis software (NIH Image 1.55, National Institute of Health, Bethesda, MD, USA), the number of pixels in the trapezoidal area under the graph curve was measured as the total setting quantity (ΔS). The ΔS value was checked for homogeneity by Bartlett's test. Analysis according to the factor of material was performed using one-way ANOVA followed by Tukey-Kramer multiple comparison test.

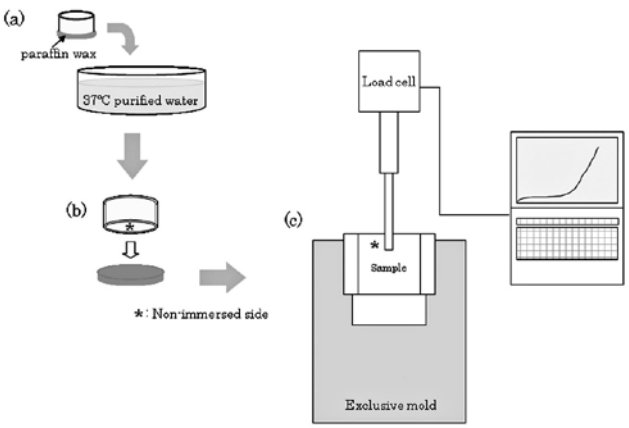

Fig. 1 Schema of setting depth measurement.
(a) Filling material was tightly filled into glass tube from the open end and immersed in distilled water.
(b) After immersion, paraffin wax was removed from the tube to expose the non-immersed side.
(c) Sample was placed on the test table so that the non-immersed side (*) was on top. Measurement needle penetrated vertically from the center of the non-immersed side to the set area. As the needle penetrated, continuous load changes were recorded using a data processing software (Trapezium, Shimadzu Corp., Japan).

Experiment 2 — Dye penetration test

For the dye penetration experiment, glass tubes with an inside diameter of 3 mm and depth of 150 mm were used. To simulate dentin, the inner surface — with which the filling would be in contact — was roughened with a carborundum stone. The rest of

the tube — except for the roughened section — was packed with cotton, and then each tube filled at the end with the test material to a depth of about 5 mm using a plastic instrument[7]. Three samples of each material were prepared for the following test conditions: Group 1 was control group with no immersion in water; Group 2 was for 30-minute immersion; Group 3 was for 60-minute immersion; and Group 4 was for 120-minute immersion.

The glass tubes in Groups 2 to 4 had their filled sections immersed in distilled water and stored in an incubator at 37ºC according to their respective setting times. Subsequently, the samples were immersed in 1% methylene blue dye solutions at 60ºC and 4ºC for two minutes each. This was repeated for 30 cycles. For Group 1, a thermal cycle test was performed immediately after the temporary filling materials were filled. After the thermal cycle test, the glass tubes were washed with adequate flowing water. The portion with the greatest dye penetration was magnified 20 times and the penetration depth was measured. Analysis according to the factors of material and immersion time was performed using two-way ANOVA followed by Tukey-Kramer multiple comparison test.

RESULTS

Setting test

The surface that contacted water exhibited expansion in all the samples. Hence, the setting depth was calculated with D_1 as a value including expansion. Table 2 shows the mean value of setting depth X of each material. When the immersion time was long, surface expansion was also large. There were some samples in which the setting depth exceeded the width of glass tubes. Figure 2 shows an example of the graph used for the analysis of total setting quantity (ΔS). Table 3 gives the mean values of ΔS in each cycle. Significant differences in ΔS were observed among the three materials (Fig. 3).

HY-Seal showed the highest setting depths at all

Table 2 Mean values of setting depth (mm)

Materials \ Day	1	2	3	4	5	6	7
Lumicon	1.39	1.55	1.86	1.95	2.06	2.37	2.61
	(0.25)	(0.15)	(0.10)	(0.18)	(0.17)	(0.24)	(0.17)
Caviton	1.18	1.77	2.59	3.62	4.28	4.67	4.90
	(0.06)	(0.22)	(0.42)	(0.83)	(0.75)	(0.37)	(0.30)
Hy-Seal	2.14	2.86	4.06	4.57	4.97	5.28	5.22
	(0.42)	(0.16)	(0.44)	(0.28)	(0.47)	(0.49)	(0.44)

( ): SD, n = 5

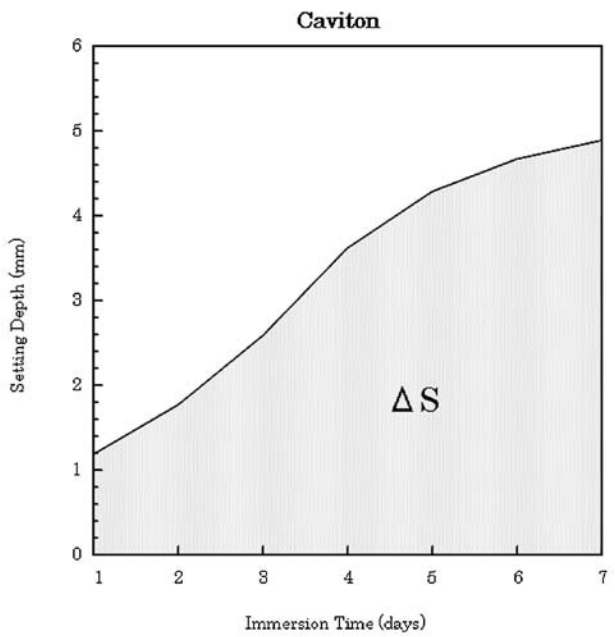

Fig. 2 Example of a graph used for the analysis of total setting quantity (ΔS).

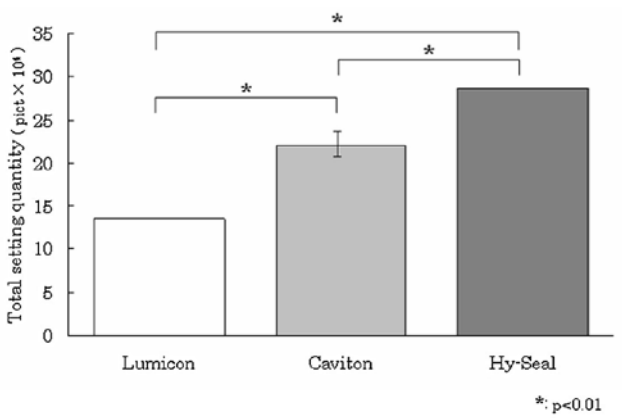

Fig. 3 Total setting quantity values of temporary filling materials (ΔS).

Table 3 Mean values of ΔS in each cycle (pict $\times 10^4$)

	cycle 1	cycle 2	cycle 3	cycle 4	cycle 5	mean
Lumicon	13.41	14.91	14.32	12.68	12.51	13.57
Cavition	20.44	22.84	22.50	21.86	22.45	22.02
Hy-Seal	30.47	28.53	29.79	26.01	28.33	28.63

Table 4 Dye penetration depth of each group

	Group 1	Group 2	Group 3	Group 4
Lumicon	0.43	0.33	0.35	0.36
	(0.11)	(0.10)	(0.04)	(0.06)
Caviton	0.97	1.36	1.39	0.85
	(0.08)	(0.12)	(0.10)	(0.06)
Hy-Seal	1.08	1.38	1.65	1.95
	(0.10)	(0.23)	(0.38)	(0.57)

( ): SD, n - 3

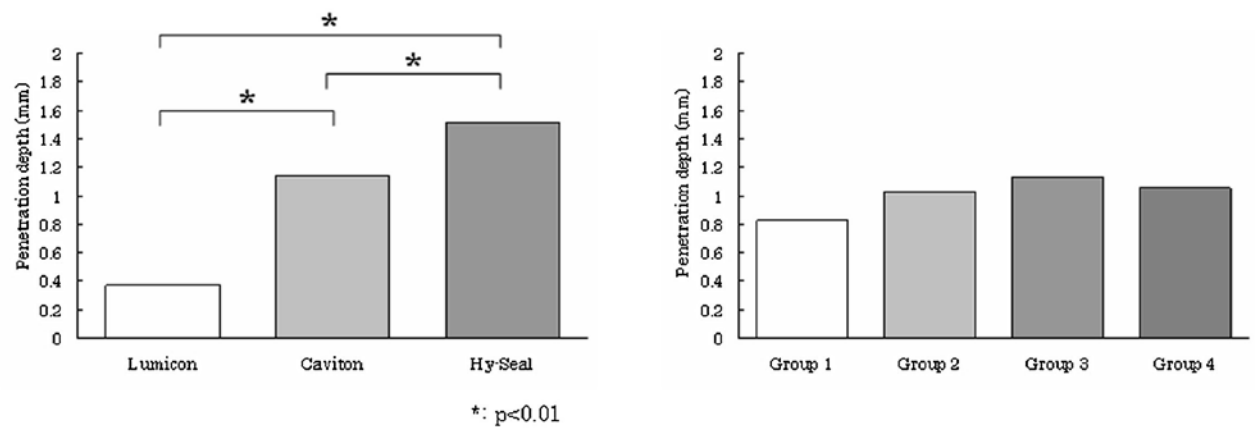

Fig. 4 Tukey's analysis of dye penetration depths of the temporary filling materials. There were significant differences between the materials ($p<0.01$)

days among the materials. After 5 days, the measurement needle could not penetrate beyond 1.5 mm because the set area had extended to almost all of the sample thickness. Caviton followed after HY-Seal, whereby penetration depth became 1 mm or less from 6 days onwards. At 7 days, setting had extended to almost all of the sample thickness for these two materials, leaving behind a minimal unset area. Comparing HY-Seal and Caviton, the penetration of the needle was deeper for HY-Seal than for Caviton due to the former's larger setting depth. These differences probably arose from their differences in surface expansion. Lumicon exhibited the smallest setting quantity. Even at 7 days, about half of the sample width was still unset.

Dye penetration test

Table 4 shows the dye penetration depth of each group. Two-way ANOVA showed significant differences between materials ($p<0.01$), but not between immersion times. Figure 4 shows the results of Tukey's analysis.

Lumicon showed the lowest penetration depth, staying constant at about 0.4 mm for each test group. With Caviton, dye penetration was 1.0 mm or less in Groups 1 and 4, but about 1.3 to 1.4 mm in Groups 2 and 3. With HY-Seal, Group 1 showed the smallest value, and that dye penetration depth increased with length of immersion time.

DISCUSSION

Temporary filling materials used for root canal procedure should possess the following requisites: (1) good sealing of the cement-tooth joint (against marginal infiltration); (2) good sealing of the cement itself (against porosity); (3) dimensional variations close to those of the tooth; (4) good resistance against abrasion and compression; (5) ease of filling and removal; (6) compatibility with the intracanal

medicaments used; and (7) good esthetic appearance[2]. Currently, zinc oxide-eugenol-based materials and resin-based materials are amongst the materials commonly used for temporary sealing. However, there is no ideal material which meets all the requisites listed above.

Hydraulic temporary sealing materials have long been in routine use because they exhibit high sealing property and are premixed for easy use with no inconsistency attributable to mixing[8]. This material sets gradually because it starts setting only when it comes in contact with water. To date, there are no standardized test methods like ISO for this material; thus, its setting time remains to be clearly elucidated. In previous studies, some test methods have been devised to investigate their setting time. For instance, Hosoya[9] attempted to investigate the setting time of hydraulic temporary sealing materials by using a Gillmore needle. It was reported that the setting time was 52 minutes at the fastest and 191 minutes at the slowest. Similarly, Isogai[10] measured the setting time with a Gillmore needle and further examined the internal conditions by cutting the specimens vertically. The shortest setting time was 26 minutes, but the sample did not set properly inside.

In the present study, the objective was to investigate the setting properties of hydraulic temporary sealing materials. To this end, the depth of the unset area was to be measured and the setting depth thereof determined by subtraction from the sample thickness. According to our experiment methodology, it was necessary for one side of each sample to be in contact with water, while the material that sealed the other end of the tube removed to measure the depth of the unset area. In our preliminary experiment, it was confirmed that sealing the glass tubes with paraffin wax prevented water penetration and precluded any undesirable influence on setting depth. Therefore, paraffin wax was used as the material to seal one end of the glass tubes in this study.

On the EZ Test device used to measure the unset area, it is a universal test machine widely used for a range of mechanical testing, such as tensile, compression, and bending tests. By using this machine with an exclusive software, continuous load changes that accompanied needle penetration through the sample were recorded. In other words, this method was simple and reliable, precluding the difficulties encountered with making indentations using Gillmore needles. However, samples were destroyed as a result of penetration by the measurement needle, making it impossible to record the continuous data of setting progress from one sample. Therefore, it was assumed that the data of one immersion cycle consisting of mean values was one set of continuous data, and ΔS values were used to compare the setting quantity.

In this study, the setting test results indicated that hydraulic temporary sealing materials set gradually from the inside of the surface where they contacted water, but the progress speed differed depending on the manufacturers. Moreover, since all the samples showed surface expansion after immersion in water, water absorption seemed to be the cause for setting expansion.

Meanwhile, exudates were observed at the other end of the glass tubes sealed with paraffin wax. This phenomenon was common to all the three materials, but the characteristics of the exudates differed. The exudate of Lumicon was yellowish, very clear and slightly viscous, while that of Caviton was cloudy and more viscous. The exudate of HY-Seal was transparent, and not very viscous. The exudates were visibly present in the samples after 2 days and tended to increase with the length of immersion time. It is suggested that as water penetrated the surface of the material and setting progressed, the viscous liquid component included in the material might be pushed to the surface of the unset area at the opposite end. Further, setting progress might have differed depending on whether the viscous liquid component could be easily substituted with water.

The sealing ability of temporary filling materials has been reported in numerous studies. Parris and Kapsimalis[11] compared the sealing properties of gutta-percha, two types of zinc phosphate temporary cements, zinc oxide-eugenol cement, Cavit, and amalgam in human extracted teeth with dye. Amongst which, Cavit showed the best sealing property which was good even when bacteria were present[12]. Lee *et al.*[3] compared the sealing properties of IRM, Cavit, and Caviton. According to their report, Caviton provided the best seal, whereby dye penetration was within the dentino-enamel junction and that expansion by water absorption during setting caused the material to adhere closely to the cavity wall. In another study by Tamse *et al.*[13], dye penetration test was conducted on two types of zinc oxide-eugenol-based materials and three types of calcium sulfate-based temporary sealing materials. It was suggested that hydrophilicity promoted dye penetration in these materials, such that amongst which calcium sulfate-based Cavidentin showed the best result. In the same vein, Webber *et al.*[14] performed a dye penetration test on Cavit. It was found that the degree of dye penetration between the temporary sealing material and dentin was about the same as that inside the material. Furthermore, it was recommended that the temporary sealing material should be at least 3.5 mm thick to prevent root canals from contamination during endodontic treatment. In the present study, the temporary

sealing materials used were also hygroscopic. Therefore, they might have permitted dye penetration not only between dentin and the materials, but also directly into the materials[15].

Prior to this experiment, dye penetration on the surface and into the temporary sealing materials was verified. Irrespective of the immersion time before thermal cycling, Lumicon showed excellent sealing with a dye penetration depth of 0.5 mm or less. With Caviton, penetration depth was 1 mm or less in Group 1 (no immersion) and Group 4 (long immersion), but increased in Groups 2 and 3. With HY-Seal, penetration depth increased with immersion time. Among the three materials tested, HY-Seal exhibited the largest extent of expansion and microcracks were also observed in some samples. At the same time, dye penetration tended to be deeper under these cracks.

Dye penetration was observed not only at the glass tube-material interface, but also into the body of the materials. A similar finding was described in previous studies[3,4,15]. However, the penetration pattern differed with materials. Whereas Lumicon and HY-Seal showed a clear boundary between the penetrated and non-penetrated portions, the boundary of Caviton became unclear gradually from the densely dyed surface to deep inside the material where pigmented spots were observed. These differences in dye penetration might be indicative of water permeation, a phenomenon which most probably accounted for the aforementioned differences in exudate characteristics observed in the setting test.

CONCLUSIONS

The results of setting and dye penetration tests were different for the three types of temporary filling materials tested, indicating that there was no correlation between setting progress and sealing ability. In other words, the sealing property of hydraulic temporary sealing materials did not seem to be improved by the setting phenomenon, but by non-hydrophobic components remaining at an unset or set area that blocked water penetration. The materials required only surface setting to withstand mechanical stresses such as mastication force, and this was sufficient to prevent deformation or destruction in the oral cavity. To improve the sealing performance effectively, it is necessary to ensure the presence of non-hydrophobic components within the temporary sealing materials to block surplus water penetration.

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