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Influence of light irradiation on Vickers hardness of dual-cure cement polymerized under restorations

Daisaku SHIOMUKI[1], Hiroyuki MINAMI[1], Takuo TANAKA[2] and Shiro SUZUKI[3]

[1] *Fixed Prosthetic Clinic, Kagoshima University Medical and Dental Hospital, 8-35-1 Sakuragaoka, Kagoshima 890-8544, Japan*
[2] *Department of Fixed Prosthetic Dentistry, Graduate School of Medical and Dental Sciences, Kagoshima University, 8-35-1 Sakuragaoka, Kagoshima 890-8544, Japan*
[3] *Department of Prosthodontics, University of Alabama at Birmingham, School of Dentistry, 1919 7th Ave. South, Birmingham, Alabama 35294-0007, USA*
Corresponding author, Hiroyuki MINAMI; E-mail: minami@dent.kagoshima-u.ac.jp

This study evaluated, by measuring Vickers hardness (Hv), the effects of these factors on the degree of polymerization of dual-cure cement (Panavia F2.0) placed under a restoration: light transmission property of restoratives materials, distance from the directly irradiated surface, and elapsed time after light irradiation. Two materials were used for the restoration: silver-palladium-copper-gold alloy (Alloy) *versus* zirconia (ZR). Restorations were cemented on bovine enamel by dual-cure cement. At 30 min, 2 h, 6 h, 1 day, and 1 week after definitive irradiation, Hv values at the enamel side of cement were evaluated at three measuring points: two points at the left and right margins and one point at the center. Data were analyzed by two-way ANOVA with Bonferroni correction (α=0.05). With the Alloy restoration, Hv value at the center was significantly lower than those at the margins at 30 min after irradiation. For both Alloy and ZR restorations, Hv value at each measuring point continued to increase significantly up to 6 h.

Keywords: Dual-cure cement, Restorative material, Light transmission property, Degree of polymerization, Vickers hardness

INTRODUCTION

In tandem with the increased use of resin cements for the placement of prosthetic restorations, their curing mode has shifted from the conventional chemical curing mode[1,2] to that of dual-curing[3,4]. An advantage of rapid initial hardening of dual-cure resin cements by light irradiation makes it easy to remove excess resin cement after restoration placement but prior to post-irradiation hardening by chemical curing, which helps to reduce clinical chairside time[5]. Compared to setting by chemical polymerization only, additional light irradiation of the exposed marginal surfaces of resin cements placed under restorations resulted in improved mechanical properties[6,7].

Numerous *in vitro* studies have shown that light irradiation improved the mechanical properties of dual-cure cements by evaluating and comparing the Knoop hardness[8,9] or tensile strengths[10,11] of these cements with and without direct exposure to curing light. However, in clinical situations, irradiated light does not directly reach the cement due to the restoration placed over the cement. To date, the degree of polymerization of dual-cure cements under restorations has not been investigated.

Studies have also shown a high correlation between the surface hardness and degree of polymerization of resins[12,13]. The purpose of this study was to investigate the degree of polymerization of a dual-cure resin cement, which was placed under a restoration and irradiated by light, by measuring its Vickers hardness (Hv). Effects of these factors on the degree of polymerization were investigated: light transmission property of restorative materials, distance from the directly irradiated surface, and elapsed time after light irradiation.

The hypotheses of this study were: (1) light transmission property of restoration would influence the Hv values of dual-cure resin cement; (2) increase in distance from the restoration margin would result in lower hardness of dual-cure resin cement; and (3) increase in elapsed time after light irradiation would cause Hv values to differ significantly among the different measuring points on resin cement.

MATERIALS AND METHODS

Metal alloy and zirconia restorations

Adherends, which mimicked restorations in this study, were fabricated from a silver-palladium-copper-gold alloy (Castwell M.C. 12, GC Corp., Tokyo, Japan; Alloy) and a zirconia block (Cercon, DeguDent GmbH, Hanau, Germany; ZR). Details of these restorative materials are given in Table 1.

Disk-shaped specimens (8×3 mm) were obtained by casting from the Alloy or by machining from the ZR block using CAD/CAM method. The bonding surface of each adherend was polished using 240-, 400-, and 600-grit silicon carbide papers (CarbiMet Abrasive Discs, Buehler, Lake Bluff, IL, USA) with ample water cooling. This was followed by airborne particle abrasion with 50-μm aluminum oxide particles (Hi Aluminas, Shofu Inc., Kyoto, Japan) at 0.4 MPa pressure for 5 s. After

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Table 1 Types of restorative materials used for adherend fabrication

Restorative material	Brand name	Composition	Manufacturer	Code
Zirconia	Cercon	ZrO_2 (89.2 wt%), Y_2O_3 (5 wt%), HfO_2 (5 wt%), *etc.*	DeguDent GmbH, Hanau, Germany	ZR
Silver-palladium-copper-gold alloy	Castwell M.C. 12	Au (12 wt%), Pd (20 wt%), Ag (46 wt%), Cu (20 wt%), *etc.*	GC Corp., Tokyo, Japan	Alloy

Table 2 Adhesive primer used for the pretreatment of alloy and zirconia surfaces

Metal primer	Composition	Manufacturer	Batch number
Alloy Primer	VBATDT, MDP, acetone	Kuraray Noritake Dental Inc.	00413A

VBATDT: 6-(4-vinylbenzyl-*n*-propyl)amino-1,3,5-triazine-2,4-dithiol, MDP: 10-methacryloyloxydecyl dihydrogen phosphate

cleaning with filtered air, all bonding surfaces were pretreated with an adhesive primer (Alloy Primer, Kuraray Noritake Dental Inc., Tokyo, Japan; Table 2), which has been shown to be effective for both Alloy[14,15] and ZR[16-18].

Enamel substrates

Labial surfaces of bovine enamel were ground using 240-, 400-, and 600-grit silicon carbide papers (CarbiMet Abrasive Discs, Buehler) to obtain flat surfaces (12×16 mm). Polishing was done using 2,000-, 4,000-, and 8,000-grit lapping films (3M™ Lapping Film, Sumitomo 3M Ltd., Tokyo, Japan) with ample water cooling.

An extremely thin layer of Vaseline with a reflective index of 1.5[19], which is similar to that of enamel of 1.6[20], was applied as a separating medium on the polished enamel surfaces. Two 100-μm-thick, black Teflon sheets (1×12 mm) were used as spacers and placed parallel to each other at 4 mm distance apart (Fig. 1). A self-etching primer (ED Primer II, Kuraray Noritake Dental Inc.; Table 3) was applied using a sponge pellet to the flat bonding surface between the spacers. Excess primer was evenly spread into a thin layer using filtered air at 30 s after application.

Bonded specimens

A dual-cure resin cement (Panavia F2.0, Kuraray Noritake Dental Inc.; Table 4) was mixed and built up on the bonding surface of restoration adherend. After the mixed cement came into contact with bovine enamel with the support and within the boundary of both spacers, a vertical load of 4.9 N was applied to the bonded specimen using a loading device for 10 s. Twenty-five specimens were each prepared for the Alloy and ZR restoration groups.

Excess resin cement which extruded out of the circumferential margins of adherend was irradiated with a light curing unit (G-Light, GC Corp.) for 5 s. The light curing unit was equipped with a 7-mm-diameter light guide tip to concentrate the light beam and was used at an irradiance of 900 mW/cm^2. Polymerized excess resin was carefully removed using a dental explorer.

Light-emitting window of the curing unit was positioned at an angle of 45 degrees to and at a distance of 3 mm from the exposed margin at each end of the

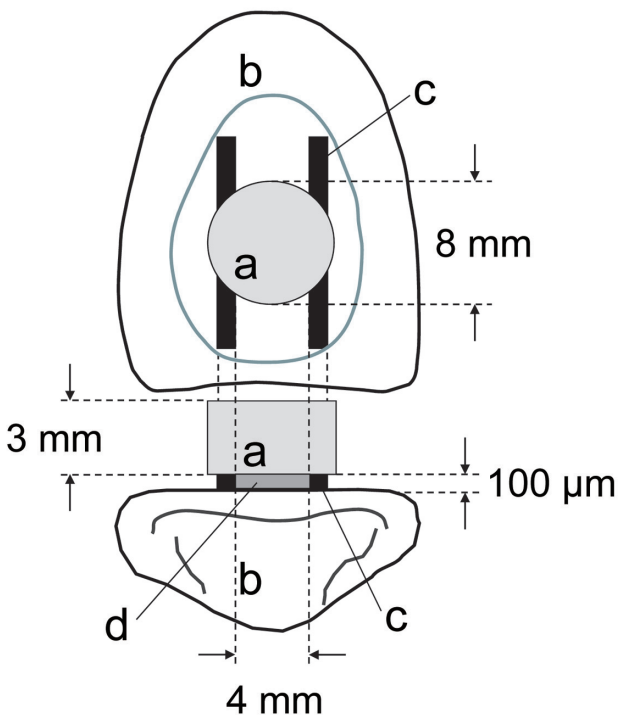

Fig. 1 Aerial (top) and lateral (bottom) views of bonded specimen, where a: disk-shaped restoration adherend; b: bovine enamel; c: spacers; and d: dual-cure resin cement.

Table 3 Chemical compositions of self-etching primer used for enamel pretreatment

Self-etching primer	Composition	Manufacturer	Batch number
ED Primer II: Liquid A	MDP, HEMA, 5-MASA, DEPT, water	Kuraray Noritake Dental Inc.	0296BA
ED Primer II: Liquid B	5-MASA, DEPT, SBSA, water	Kuraray Noritake Dental Inc.	0170AA

MDP: 10-methacryloyloxydecyl dihydrogen phosphate, HEMA: 2-hydroxyethyl methacrylate, 5-MASA: *N*-methacryloyl-5-aminosalicylic acid, DEPT: *N*,*N*-di(hydroxyethyl)-p-toluidine, SBSA: Sodium benzene sulfinate

Table 4 Chemical compositions of dual-cure resin cement used for bonding restoration adherends to bovine enamel

Resin cement	Composition	Manufacturer	Batch number
Panavia F2.0: Paste A	MDP, Bis-MPEPP, hydrophobic and hydrophilic DMAs, BPO, CQ, silica filler	Kuraray Noritake Dental Inc.	0501AA
Panavia F2.0: Paste B	Bis-MPEPP, hydrophobic and hydrophilic DMAs, DEPT, sodium 2,4,6-TPBSA, silica filler, barium glass filler, TiO_2, NaF	Kuraray Noritake Dental Inc.	0114AA

MDP: 10-methacryloyloxydecyl dihydrogen phosphate, Bis-MPEPP: 2,2-bis[(4-methacryloxy polyethoxy)phenyl]propane, DMA: Dimethacrylate, BPO: Benzoyl peroxide, CQ: dl-camphorquinone, DEPT: *N*,*N*-di(hydroxyethyl)-p-toluidine, Sodium 2,4,6-TPBSA: Sodium 2,4,6-triisopropyl benzene sulfinate, NaF: Sodium fluoride

resin cement, which was located under the restoration and between the spacers. After definitive irradiation was directed at both exposed cement margins for 20 s each, bonded specimens were immediately immersed in 37°C distilled water in a shaded incubator.

Vickers hardness measurement

At 30 min, 2 h, 6 h, 1 day, and 1 week after definitive irradiation, adherends were carefully detached from bovine enamel. Five specimens each from Alloy and ZR restoration groups were assigned for each of the five elapsed time intervals.

On the enamel side of resin cement (*i.e.*, side of resin cement bonded to bovine enamel), Vickers hardness was measured using a microhardness tester (HM-102-SM, Mitutoyo Corp., Akashi, Kanagawa, Japan). A load of 200 g was applied for 15 s, and both indentation diagonals (mm) were measured. Vickers hardness (Hv) was determined using the equation, $HV=1.8544\ F/d^2$, where F is the indentation load (kgf) and d is the arithmetic mean of two diagonals (mm).

Measurements were performed at three measuring points: two points A and C were near both ends of the exposed cement margins, and point B was at the center of resin cement (Fig. 2). Hv values of Alloy and ZR restoration groups at 30 min after light irradiation were used as controls.

Statistical analysis

Mean Hv values were determined at each measuring point for each elapsed time interval. Statistical analysis was performed using three-way analysis of variance (ANOVA) with the adherend material, measuring point, and elapsed time interval as independent factors. Multiple comparisons were performed using the Bonferroni-Dunn test to identify significant differences among the groups ($\alpha=0.05$).

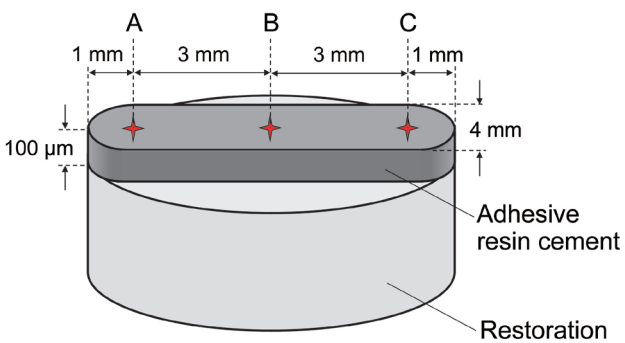

Fig. 2 Three Vickers microhardness measuring points on polymerized resin cement, where A and C: near either end of exposed cement margins; B: center of resin cement.

RESULTS

Three-way ANOVA results revealed that there were significant differences in Hv value between the two restoration adherend materials ($p=0.006$) and among the five elapsed time intervals ($p<0.001$). However, there were no significant differences among the three measuring points ($p=0.308$). There was also a significant

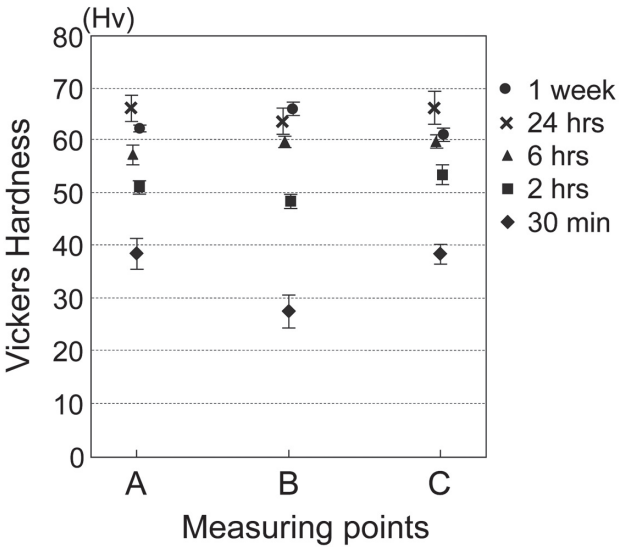

Fig. 3 Hv results at each measuring point and elapsed time interval for Alloy specimens.
Error bars depict standard deviations.

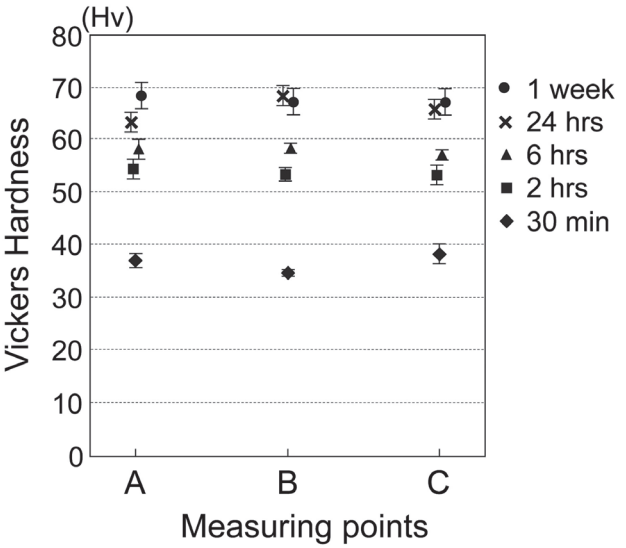

Fig. 4 Hv results at each measuring point and elapsed time interval for ZR specimens.
Error bars depict standard deviations.

interaction among these three factors (p=0.04).

Vickers hardness of Alloy restorative material

Figure 3 shows the Hv results of Alloy adherend group. At 30 min, Hv values of points A and C were significantly higher than that of point B. However, at 2 h and beyond, no significant differences were observed among the three measuring points.

At each measuring point, Hv value at 2 h was significantly higher than that at 30 min. At point B, Hv value at 6 h was significantly higher than that at 2 h. At point A, Hv value at 1 day was significantly higher than that at 6 h. Between 1 day and 1 week, no significant differences were observed at each measuring point.

Vickers hardness of ZR restorative material

Figure 4 shows the Hv results of ZR adherend group. At each elapsed time interval, there were no significant differences in Hv value among all the three measuring points.

At each measuring point, Hv value at 2 h was significantly higher than that at 30 min. Between 6 h and 1 day, Hv value at 1 day was significantly higher than that at 6 h at point C. Between 2 h and 6 h and between 1 day and 1 week, no significant differences were observed at each measuring point.

DISCUSSION

The hypotheses of this study were partially accepted. At 30 min after definitive irradiation, Hv values at both ends of cement margins were significantly higher than that at the center for Alloy adherend group. As elapsed time increased from 30 min to 2 h, Hv values significantly increased at each measuring point for both Alloy and ZR adherend groups. However, between 1 day and 1 week, no significant differences were observed at all measuring points for both adherend groups.

For measuring points A and C which were located at both ends of cement margins, no significant differences in Hv value were observed these two measuring points at each elapsed time interval for both Alloy and ZR adherend groups. Therefore, light irradiation conditions at measuring points A and C were consistent and the same, thus paving the way for comparison between the central area of resin cement *versus* the cement margins.

Choices of materials used in this study

Initially, it was intended that all materials and methods used in this study would completely simulate those in a clinical setting. This meant that bonding surfaces of adherends were pretreated with airborne particle abrasion and MDP-containing primer. Before restoration was bonded to the enamel of abutment tooth, phosphoric acid etching[21] —not self-etching primer containing a polymerization accelerator— was applied to the abutment tooth. Therefore in the preliminary study, Hv values were measured without prior application of self-etching primer on bovine enamel.

It was also assumed that by merely irradiating the restoration margins, irradiated light could partially reach the central area under the restoration by reflection and refraction of light on the enamel. The outcome was such that dual-cure resin cement placed under the Alloy restoration was insufficiently polymerized and remained in a gel state at 30 min after light irradiation. This was because irradiated light at restoration margins could not reach the middle region of resin cement. As a result, no

indentations were made and no Hv measurements were carried out in the preliminary study.

In the present study, Vaseline was used as a separating medium. In the preliminary study, 50-μm-thick polyester film (Lumirror T60, Toray Industries, Inc., Tokyo, Japan) was tested as a separating medium. At two hours after light irradiation and thereafter, there were no significant differences in Hv value between the use of polyester film or Vaseline. This meant that Vaseline did not affect the polymerization of the primer or resin cement. For this reason and in compliance with the clinical practice of phosphoric acid etching of enamel, a separating medium was finally employed on bovine enamel surfaces prior to the application of a self-etching primer[22] (Table 3) which contained DEPT as a polymerization accelerator[23]. Application of the self-etching primer also improved the polymerization of dual-cure cement in areas covered by the restoration. As a result, at 30 min after light irradiation, dual-cure resin cement located under the center of Alloy restoration was sufficiently polymerized for Hv measurements to be carried out in the current study.

Vickers hardness of resin cement placed under Alloy restoration

At 30 min after light irradiation, Hv values at cement margins (measurement points A and C) were significantly higher than that at the central area (measurement point B) (Fig. 3). The Alloy material could not transmit light, and light irradiated at the margins could not reach the central area. If irradiated light were to reach the central area by reflection or refraction, intensity of the transmitted light would be rather low. Therefore, the central area of dual-cure cement was considered to set mainly by chemical polymerization. In contrast, both chemical polymerization and photopolymerization modes progressed simultaneously at the cement margins due to their direct exposure to the curing light. As a result, the cement margins yielded higher Hv values than the central area.

In the present study, additional light irradiation was performed after removal of polymerized excess cement. If this were not performed, the cement margins might also yield low Hv values like the central area, consequently resulting in inferior bonding of prostheses. Therefore, when installing a restoration on abutment tooth using dual-cure resin cement in a clinical setting, it is highly recommended to perform additional light irradiation at restoration margins to accelerate cement curing.

At 2 h and beyond after light irradiation, no significant differences in Hv value were observed between the central area and both ends of cement margins. This implied that hardening by chemical polymerization progressed rapidly from 30 min to 2 h after irradiation, such that Hv value achieved solely by chemical polymerization (at the central area) caught up with that acquired by dual curing modes (at the cement margins). In other words, when dual-cure resin cement was polymerized solely by chemical polymerization, it required 2 h to achieve the same hardness as that acquired by dual-cure mode.

Vickers hardness of resin cement placed under ZR restoration

At 30 min after light irradiation, no significant differences in Hv value were observed between the central area (measurement point B) and both ends of cement margins (measurement points A and C) (Fig. 4). This result was probably due to high light transmission through the ZR adherend. It was reported that the polymerization efficiency of dual-cure resins was affected by numerous factors: irradiation duration[24], light intensity[25], light transmission[26,27], and ceramic shade[26] and thickness[28,29]. Specifically, a translucent ceramic which allowed more light to transmit through the material, as compared to an opaque ceramic, resulted in improved mechanical properties of dual-cure resin cement placed under the restorative material[30].

In a study by Turp *et al.*[27], dual-cure resin cement was polymerized by light irradiation through 2 mm-thick bi-layered ceramic disks (1.0-mm-thick feldspathic porcelain on top of 1.0-mm-thick ZR). When restoration thickness exceeded 2 mm, a longer light irradiation time than that recommended by the manufacturer was needed[27]. In another study by Kilinc *et al.*[26], overlying ceramic thickness of 3 mm and above was found to adversely affect the polymerization efficiency of dual-cure resin cements. Therefore, 3-mm ceramic thickness was considered as the critical threshold[26].

In the present study, the use of 3-mm-thick ZR restorations could be deemed comparatively thick in consideration of the restoration thicknesses selected and employed in other studies[26,27,30]. Nonetheless, the high light intensity of the light curing unit (900 mW/cm^2)[25], coupled with the high light transmission property of ZR[27], enabled the polymerization of dual-cure resin cement placed under the ZR restoration.

Clinical significance

At 6 h and beyond after light irradiation, there were no significant increases in Hv value for both Alloy and ZR adherend groups. Nonetheless, Hv values at 1 day after irradiation were approximately twice those at 30 min after irradiation. This implied that the chemical polymerization process of dual-cure resin cement did not complete within a short period, but lasted considerably longer as in the case of conventional resin cements which hardened solely by chemical polymerization[6,7].

It is highly recommended to keep to a minimum stresses on the bonding interface the day that a prosthetic restoration is installed[31]. Examples of stress-inducing activities include polishing and removal of excess resin cement. Results of this study supported these clinically relevant recommendations. Finishing procedures should be carried out at least 1 day after light irradiation.

Limitations of present study

There were several limitations in the simulation of clinical situations in this study. They pertained to irradiation duration, light intensity, light transmission

property of restorative materials, restoration thickness and shade. These factors influenced the polymerization efficiency of dual-cure resin cements, but which were not adequately simulated and evaluated in this study. Therefore, further studies —which better simulate these clinical situations— are necessary for a thorough evaluation of the polymerization degree of dual-cure resin cements placed under restorations.

CONCLUSIONS

This *in vitro* study evaluated the degree of polymerization of a dual-cure resin cement placed under two restorative materials with different light transmission properties. Degree of polymerization was evaluated by measuring Hv values as a function of elapsed time after definitive irradiation. Within the limitations of this study, the following conclusions were drawn:

1. When placed under the Alloy restoration, Hv value of resin cement at the center was significantly lower than those at cement margins at 30 min after light irradiation. However, there were no significant differences among all measuring points at 2 h and thereafter.
2. When placed under the ZR restoration, no significant differences in Hv value were observed among the central measuring point and those at cement margins at 30 min after light irradiation.
3. Irrespective of restorative material, the Hv values of both measuring points at cement margins —which were directly exposed to curing light— increased significantly up to 2 h after light irradiation.
4. For both restorative materials, Hv values of resin cement at the center and both ends of cement margins continued to increase significantly up to 6 h after light irradiation. At 6 h, 1 day, and 1 week after light irradiation, no significant differences in Hv value were found among these elapsed time intervals at each measuring point, except between 6 h and 1 day at one ends for both Alloy and ZR restoration groups.

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