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# Simulated localized wear of resin luting cements for universal adhesive systems with different curing mode

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**Abstract:** This study evaluated the simulated localized wear of resin luting cements for universal adhesive systems using different curing modes. Five resin luting cements for universal adhesive systems were evaluated and subsequently subjected to wear challenge in a Leinfelder-Suzuki wear simulation device. Overall, 20 specimens from each resin luting cement were photo-cured for 40 s (dual-cure group), and 20 specimens of each material were not photo-cured (chemical-cure group). Simulated localized wear was generated using a stainless steel ball-bearing antagonist in water slurry of polymethylmethacrylate beads. In addition, scanning electron microscopy (SEM) observations of resin luting cements and wear facets were conducted. Significant differences in simulated wear and SEM observations of wear facets were evident among the materials in the dual- and chemical-cure groups. The simulated wear and SEM observations of wear facets of G-CEM LinkForce and Panavia V5 were not influenced by the curing mode.

SEM observations of resin luting cements were material dependent. In most cases, dual curing appears to ensure greater wear resistance of resin luting cements than chemical curing alone. The wear resistance of some resin luting cements appears to be material dependent and is not influenced by the curing mode.

Keywords: simulated localized wear; resin luting cement; curing mode.

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## Introduction

Increasing aesthetic demands in clinical dentistry have resulted in continued advances and development of ceramic restorations (1), which include intra- and extra-coronal restorations that require utilization of luting cements (2). Several issues associated with the fit and marginal adaptation of ceramic restorations seem to significantly influence their durability (3). Therefore, selection of effective luting cement is important for the long-term clinical success of these restorations (4). The durability of ceramic restorations has been increased using a resin luting cement with high-mechanical and low-solubility properties (5). Despite their improvement in physical properties, frequent interfacial and/or marginal defects have been clinically observed around bonded ceramic restorations (6). Surface hardness and wear resistance of resin luting cements are essential

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**Table 1** Resin luting cements for universal adhesive systems

Resin luting cement (Code, shade)	Main components (Filler content)	Manufacturer (Lot No.)
Calibra Esthetic Resin Cement (CE, yellow)	Dimethacrylate resins, Glass fillers, Fumed silica, Initiators, Stabilizers, Pigments (67.4 wt%, 45.3 vol%)	DENTSPLY Caulk, Milford, DE, USA (13394)
G-CEM LinkForce (GL, A2)	Dimethacrylate, Silica filler, Initiators, Stabilizers, Pigments (63.0 wt%, 38.0 vol%)	GC Corporation, Tokyo, Japan (1407281)
Multilink Automix (MA, yellow)	Dimethacrylate, HEMA, Barium glass filler, Silica filler, Ytterbium trifluoride, Initiators, Stabilizers, Pigments (68.5 wt%, 42.5 vol%)	Ivoclar Vivadent, Schaan, Lichtenstein (150317)
Panavia V5 (PV, Universal)	Bis-GMA, TEGDMA, Hydrophobic aromatic dimethacrylate, Hydrophilic aliphatic dimethac- rylate, Barium glass filler, Fluoroaluminosilicate glass, Silica filler, Initiators, Stabilizers, Pigments (61.0 wt%, 38.0 vol%)	Kuraray Noritake Dental, Tokyo, Japan (A90026)
RelyX Ultimate Adhesive Resin Cement (RU, A1)	Methacrylate monomer, Alkaline filler, Initiator components, Stabilizers, Pigments, Rheological additives, Fluorescence dye (67.0 wt%, 43.0 vol%)	3M ESPE, St. Paul, MN, USA (588835)

HEMA, hydroxyethyl methacrylate; Bis-GMA, bisphenol A glycidyl methacrylate; TEGDMA, triethylene glycol dimethacrylate

material properties to overcome the abovementioned issues (7). While the wear characteristics of restorative materials have been extensively examined (8-11), limited research is available in the area of resin luting cements.

Controlled clinical studies on ceramic inlay and onlay restorations using resin luting cements revealed significant differences in marginal integrity between the baseline and 5- or 12-year recall evaluation (12,13). This is particularly important for inlay and onlay restorations of posterior teeth, where the cement margin is exposed on the occlusal surface. In clinical situations, occlusal stress is transmitted to the resin luting cement layer, particularly in filler-resin matrix interface, during both functional and parafunctional activities (14). Stress concentrations at the filler-resin matrix interface may result in filler dislodgement and exposure of the resin matrix, leading to wear (15). Therefore, the wear characteristics of resin luting cements remain a clinical concern, particularly for ceramic restorations in occlusal contact areas.

Recently, resin luting cements for use with universal (10-methacryloyloxydecyl dihydrogen phosphate-based) adhesive systems have been introduced (16). Universal adhesive systems can be used in total-etch, self-etch, or selective-etch modes (17) and can be used to bond various substrates in addition to tooth substrate (18). The physical properties of resin luting cements have been improved using nano-sized fillers and modifying the resin matrix formulation (19). The versatility of universal adhesive systems and improved mechanical properties of the resin luting cement offer a new and simplified approach to

bonding ceramic restorations to teeth (20,21). Since the recent development of resin luting cements for universal adhesive systems, little information is currently available about their wear properties.

In the occlusal contact area, wear is caused by the contact with the opposing tooth and is considered as a localized process mainly related to local microfracture (22). Latta et al. developed a laboratory simulated model for evaluation of localized wear that simulates masticatory stresses to a specimen through a stainless steel conical stylus in the presence of slurry of polymethylmethacrylate (PMMA) beads (23). Therefore, the simulation of localized wear has facilitated the development of *in vitro* studies than can help predict a material's performance *in vivo*. A previous study that compared clinical data of two resin composite materials from two study sites indicated a close relationship between simulated localized wear in the laboratory and clinical wear in the occlusal contact area (24).

This study investigated the simulated localized wear of resin composites for universal adhesive systems using different curing modes. The null hypothesis to be tested was that the simulated localized wear of resin luting cements for universal adhesive systems would not be influenced by the type of material or the curing mode.

## Materials and Methods

### Study materials

Five resin luting cements for universal adhesive systems were evaluated in this study: Calibra Esthetic Resin

Cement (CL, DENTSPLY Caulk, Milford, DE, USA), G-CEM LinkForce (GL, GC, Tokyo, Japan), Multilink Automix (MA, Ivoclar Vivadent, Schaan, Liechtenstein), Panavia V5 (PV, Kuraray Noritake Dental, Tokyo, Japan), and RelyX Ultimate Adhesive Resin Cement (RU, 3M ESPE, St. Paul, MN, USA). The associated lot numbers, components, and percentages of filler loading in the five resin luting cements are listed in Table 1.

### Specimen preparation

Custom stainless steel fixtures containing a cylindrical cavity (diameter, 6.5 mm; depth, 4.0 mm) were machined for localized wear testing. Twenty specimens (designated as the dual-cure group) of each resin luting cement were photo-cured in two increments each of approximately 2 mm depth with a quartz-tungsten-halogen unit (Spectrum 800 Curing Unit, DENTSPLY Caulk) set at 600 mW/cm<sup>2</sup> for 40 s at a standardized distance of 2 mm. Twenty specimens (designated as the chemical-cure group) were not photo-cured. After water storage for 24 h at 37°C, the cement surfaces of dual- and chemical-cure groups were polished flat to 4,000 grit using a sequence of silicon carbide papers (Struers, Cleveland, OH, USA) and a grinder-polisher (Ecomet 4, Buehler, Lake Bluff, IL, USA).

### Localized wear simulation

A Leinfelder-Suzuki (Alabama) device was used for wear simulation. The simulator had a plastic water bath, and the custom wear fixtures were mounted inside the four-station bath. A brass cylinder was then placed around each fixture in the bath to serve as a reservoir for the abrasive medium (i.e., water slurry of unplasticized PMMA with an average particle size of 44 μm). The medium was placed inside the brass cylinders to cover the surface of the resin luting cements in the custom fixtures. The PMMA water slurry inside the brass cylinders was approximately 6.0 mm thick over the surface of the resin cement.

A stainless steel ball bearing (radius, 2.387 mm) was mounted inside a collet assembly for the localized wear simulations. The antagonist tips were mounted on spring-loaded pistons to deliver the wear challenges. During load application, the antagonists rotated approximately 30° when maximum force was reached (maximum load of 78.5 N at a rate of 2 Hz) and then counter-rotated back to the original starting position as the load was relaxed to complete the cycle. In addition, 400,000 cycles were used for localized wear simulation.

### Localized wear measurement

Prior to the wear simulation, each resin luting cement specimen was profiled with a ProScan 2100 non-contact optical profilometer (Scantron Industrial Products, Taunton, UK) using the ProScan software. These profiles provided the pre-test digitized surface contours (20 specimens from each of the five resin luting cements in dual- and chemical-cure groups were used for the simulated localized wear tests).

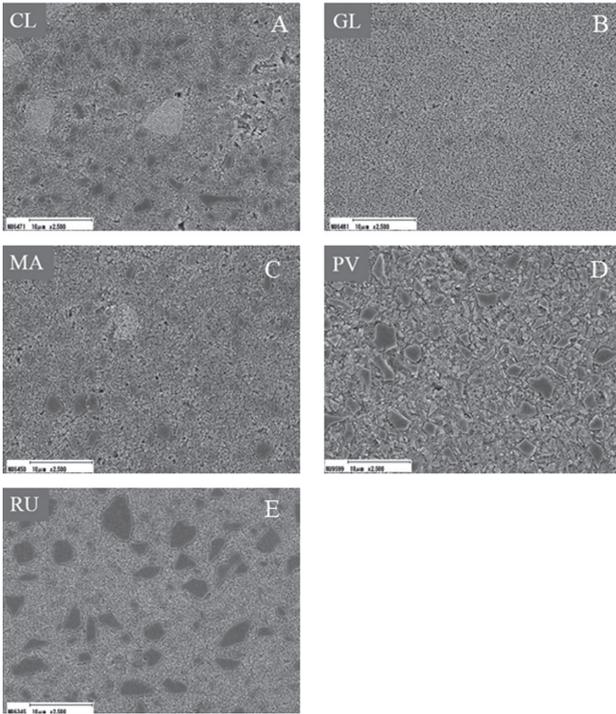
After localized wear simulation, the specimens were ultrasonically cleaned (L&R T-14B solid state ultrasonic cleaner, L&R Manufacturing Company, South Orange, NJ, USA) in distilled water for 3 min and then profiled again with the ProScan 2100 unit. The X, Y, and Z coordinates of the “before” and “after” scans were exported from the ProScan software to another computer for analysis using the AnSur 3D software (Minnesota Dental Research Center for Biomaterials and Biomechanics, University of Minnesota, Minneapolis, MN, USA).

Volume loss (VL, mm<sup>3</sup>) and maximum depth (MD, μm) of the wear facets on the specimens were determined from the differences between the “before” and “after” data sets. A computerized fit of the data sets was first completed with the AnSur 3D software. VLs and MDs of the facets were subsequently determined for each of the five resin luting cements polymerized in the dual-cure and chemical-cure modes.

### Scanning electron microscopy (SEM) observations of the resin luting cement surfaces

The ultrastructure of the polished surfaces of the resin luting cements was observed after argon-ion etching. Three specimens per group were observed using field-emission SEM (ERA 8800FE, Elionix, Tokyo, Japan).

The surfaces of the resin luting cements were polished flat to 4,000 grit using a sequence of silicon carbide papers and a grinder-polisher. The surfaces were subsequently polished using abrasive disks (Fuji Star Type DDC, Sankyo Rikagaku, Okegawa, Japan) followed by a series of diamond pastes down to a particle size of 0.25 μm (DP-Paste, Struers) to obtain a high-gloss finish. To enhance the visibility of the filler particles, the polished surfaces were etched for 30 s with argon-ion beams (EIS-200ER, Elionix) directed perpendicular to the surfaces at an accelerating voltage of 1.0 kV and with a current density of 0.4 mA/cm<sup>2</sup>. Next, the surfaces were coated with a thin film of gold in a vacuum evaporator (Quick Coater SC-701, Sanyu Electron, Tokyo, Japan). An operating voltage of 10 kV was used for SEM observations.



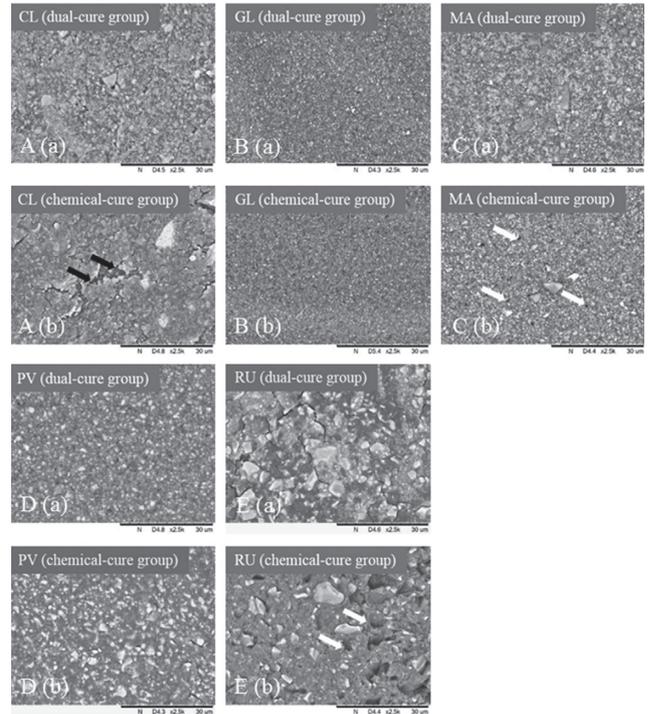
**Fig. 1** Representative scanning electron microscopy images of polished resin luting cement surfaces. The larger size ranges of irregular particles in the cements are as follows: A. CL (0.1-8  $\mu\text{m}$ ), B. GL (0.1-1.0  $\mu\text{m}$ ), C. MA (1-4  $\mu\text{m}$ ), D. PV (0.1-6  $\mu\text{m}$ ), and E. RU (0.1-10  $\mu\text{m}$ ). Filler sizes and distribution in the different types of resin luting cements varied depending on the material. CL, Calibra Esthetic Resin Cement; GL, G-CEM LinkForce; MA, Multilink Automix; PV, Panavia V5; RU, RelyX Ultimate Adhesive Resin Cement. 2,500 $\times$  magnification.

### SEM observations of the wear facets

The ultrastructure of representative wear facets of the resin luting cements from both the dual- and chemical-cure groups for localized wear simulations were observed using SEM (TM3000 TableTop SEM, Hitachi-High Technologies, Tokyo, Japan). After the wear analysis, representative specimens were coated with a thin film of gold-palladium in a vacuum evaporator (Emitech SC7620 Mini Sputter Coater, Quorum Technologies, Ashford, UK). An operating voltage of 15 kV was used for SEM observations.

### Statistical analyses

VLs and MDs of the wear facets on the resin luting cements were analyzed using a commercial statistical software package (SPSS Statistics Base, International Business Machines, Armonk, NY, USA). Two-way analysis of variance (ANOVA) and Tukey's *post-hoc* tests were used to analyze each data set, with a significance level of  $\alpha = 0.05$ .



**Fig. 2** Representative scanning electron microscopy (SEM) images of the wear facets on resin luting cements. SEM images of the worn surfaces of CL, MA, and RU in the chemical-cure group show higher degrees of cracking (black arrows) or filler particle plucking (white arrows) than those in the dual-cure group. On the contrary, SEM images of the wear facets of GL and PV samples did not exhibit any clear differences between the two curing modes. A. CL, Calibra Esthetic Resin Cement; B. GL, G-Cem LinkForce; C. MA, Multilink Automix; D. PV, Panavia V5; E. RU, RelyX Ultimate Adhesive Resin Cement. 2,500 $\times$  magnification: (a) dual-cure group and (b) chemical-cure group.

## Results

### Localized wear simulation

The VL and MD results for the wear facets of the resin luting cements for universal adhesive systems after the localized wear simulations are shown in Tables 2 and 3, respectively. Two-way ANOVA results indicated that material type ( $P < 0.001$ ), curing mode ( $P < 0.001$ ), and the interaction between the two ( $P < 0.001$ ) significantly affected the VLs and MDs of the five resin luting cements.

Significant differences were evident among the materials in the dual- and chemical-cure groups. Similar trends were observed in the VLs and MDs of the dual- (GL < MA < PV < CL < RU) and chemical- (GL < PV < MA < CL < RU) cure groups following localized wear simulations. Unlike CL, MA, and RU, the VLs and MDs of the wear facets of GL and PV were not influenced by the curing mode.

### SEM observations of the resin cement surfaces

Representative SEM images of the polished resin luting

cement surfaces are shown in Fig. 1A-E. Argon-ion etching revealed clear differences in filler particle size, shape, and distribution in the specimens studied. The resin luting cement specimens exhibited various filler particle sizes and shapes.

SEM images of the polished and argon-etched CL surfaces revealed the presence of irregular particles with sizes ranging from 0.1-8  $\mu\text{m}$  (Fig. 1A). However, SEM images of polished and argon-etched GL surfaces (Fig. 1B) showed smaller irregular particles (0.1-1.0  $\mu\text{m}$ ). Irregular particle sizes of 0.1-4  $\mu\text{m}$ , 0.1-6  $\mu\text{m}$ , and 0.1-10  $\mu\text{m}$  were observed in SEM images from the MA (Fig. 1C), PV (Fig. 1D), and RU (Fig. 1E) specimens, respectively.

### SEM observations of the wear facets

Representative SEM images of wear facets that were obtained after conducting the localized wear simulations in the dual- and chemical-cure groups are shown in Fig. 2 A-E. SEM images of the worn surfaces of CL (Fig. 2Ab), MA (Fig. 2Cb), and RU (Fig. 2Eb) in the chemical-cure group appeared to indicate a higher extent of cracking or filler particle plucking after performing localized wear simulations compared with the specimens in the dual-cure group (Fig. 2Aa, 2Ca, 2Ea). Conversely, SEM images obtained after localized wear simulation of the worn surfaces of GL (Fig. 2Ba, 2Bb) and PV (Fig. 2Da, 2Db), which were polymerized using the two different curing modes, did not exhibit any significant differences.

## Discussion

Typically, the wear resistance of restorative materials is evaluated by laboratory methods involving the use of an electric device that repeatedly makes contact with the material being tested, utilizing an antagonist object such as a stylus (25). In 2001, the International Organization for Standardization published a technical specification, "Wear by two- and/or three-body contact," describing eight laboratory methods (26). One of these methods was the Alabama method, which was developed at the University of Alabama (22). Barkmeier et al. (27) have recently developed the simulated localized wear, which was used by Latta et al. (23), using a ProScan 2100 non-contact profilometer in conjunction with the AnSur 3D software for wear analysis. In the present study, this method was used to measure the simulated localized wear in the resin luting cements.

Simulated localized wear of resin luting cements for universal adhesive systems in the dual- and chemical-cure groups clearly showed significant differences among the different materials used. The filler load was reported to

play a particularly important role in the wear resistance of resin-based materials, wherein higher filler loads have been shown to reduce the level of wear (5). The filler load values provided by the manufacturers varied greatly between the resin luting cements in the present study (Table 1), and the rank order of the filler load values of the tested resin luting cements in wt% (MA > CE > RU > GL > PV) and vol% (CE > RU > MA > GL = PV) were different from the simulated wear of both the dual-cure (GL < MA < PV < CL < RU) and chemical-cure (GL < MA < PV < CL < RU) groups. Therefore, in this study, the filler load values did not seem to directly influence the simulated localized wear of resin luting cements. As simulated wear is affected by many factors including the type and content of the resin matrix, surface treatment of the filler, and polymerization kinetics, a relatively weak association with a single factor is not surprising. Available information about the detailed compositional content of the tested resin luting cements, technologies used for the surface treatment of the filler, and polymerization kinetics is so modest that no further comparison can be made. Conversely, a clear relationship between simulated wear and filler size was found in the present study. The trend of filler particle size among the polished resin luting cements (GL < MA < PV < CL < RU), as determined from the SEM observations using argon-ion etching, was the same as that for the simulated localized wear models in the dual-cure group (GL < MA < PV < CL < RU). Furthermore, the results of our study are in agreement with a previous study that reported that the filler particle size affects the simulated wear of resin composites (28). Resin composites with smaller-sized fillers showed lower simulated wear. An example of this is GL from the dual-cure group that had smaller filler particles and produced less simulated wear. On the contrary, RU, which had larger filler particles with a wide size distribution, exhibited greater wear regardless of the wear simulation model. Therefore, the simulated localized wear of the resin luting cements was influenced by filler particle size, as in the case of resin composites (28). Differences between the wear resistances of the different types of resin luting cements have been hypothesized to result from the lower inter-particle spacing between the small filler particles. Given that small filler particles are more closely packed, the resin matrix between them is protected from further wear (29). By contrast, when larger filler particles are dislodged from the resin composite surface, a void is produced, thereby exposing the underlying resin matrix to wear (30). In addition, the removed particles can cause further abrasion on the surfaces of the resin-based materials, leading to increased wear (31).

**Table 2** Simulated localized wear of resin luting cements volume loss (VL)

Resin luting cement	VL (mm <sup>3</sup> )		
	Dual-cure group	Chemical-cure group	Difference
CL	0.116 (0.016) <sup>a,c,A</sup>	0.160 (0.020) <sup>a,B</sup>	0.044
GL	0.078 (0.020) <sup>b,A</sup>	0.089 (0.018) <sup>b,B</sup>	0.011
MA	0.095 (0.017) <sup>c,A</sup>	0.136 (0.026) <sup>a,B</sup>	0.041
PV	0.103 (0.024) <sup>c,A</sup>	0.120 (0.026) <sup>a,A</sup>	0.017
RU	0.126 (0.017) <sup>a,A</sup>	0.205 (0.016) <sup>c,A</sup>	0.079

Values in parenthesis are standard deviations. Same small-case letter in the same vertical column indicates no significant difference ( $P > 0.05$ ). Same upper-case letter within individual rows indicates no significant difference ( $P > 0.05$ ).

**Table 3** Simulated localized wear of resin luting cements maximum depth (MD)

Resin luting cement	MD ( $\mu\text{m}$ )		
	Dual-cure group	Chemical-cure group	Difference
CL	181.1 (12.1) <sup>a,A</sup>	206.7(11.4) <sup>a,B</sup>	25.6
GL	152.2 (16.5) <sup>b,A</sup>	162.7 (15.1) <sup>b,B</sup>	10.2
MA	167.4 (12.1) <sup>b,A</sup>	199.3 (16.1) <sup>a,B</sup>	32.9
PV	174.9 (14.5) <sup>a,b,A</sup>	189.8 (22.8) <sup>a,A</sup>	14.9
RU	187.4 (10.2) <sup>a,A</sup>	237.4 (13.2) <sup>c,A</sup>	50.0

Values in parenthesis are standard deviations. Same small-case letter in same vertical column indicates no significant difference ( $P > 0.05$ ). Same upper-case letter within individual rows indicates no significant difference ( $P > 0.05$ ).

Although different types of resin luting cements with similar shades should ideally be used for a comparative study, the types of resin luting cements used varied in shades and translucencies (Table 1) because of the limited number of shades and different color systems of each manufacturer. Moreover, the shades and translucencies of resin-based materials have been reported to significantly influence their degrees of conversion (32). Therefore, the varied shades and translucencies of the resin luting cements in the present study may have some influences in their values of the simulated localized wear.

The simulated localized wear of the resin luting cements differed significantly depending on the curing mode in this study. As shown in Tables 2 and 3, the trend observed for the simulated localized wear in the chemical-cure group (GL < PV < MA < CL < RU) was different from that seen in the dual-cure group (GL < MA < PV < CL < RU). Previous studies evaluating the influence of the curing method on the degree of conversion of resin luting cements have generally found that dual curing produces a significantly higher degree of conversion than chemical curing alone (33,34). In addition, the wear resistances and mechanical properties of resin-based materials reportedly increased by improving the degree of conversion (35,36). The ability of dual-cure resin luting cements to cure effectively in the chemical-curing mode is a key to the long-term clinical success of restorations, particularly when dual curing is not possible

or is limited. Therefore, on the basis of the results of this study, clinicians should pay more attention to the different setting characteristics of dual-cure resin luting cements when selecting these materials for clinical use.

The VLs and MDs of RU, MA, and CL significantly increased in the chemical-cure group compared with the dual-cure group. However, the VLs and MDs of GL and PV were not influenced by curing mode. SEM images of the worn surfaces exhibited increased cracking and filler particle plucking in the CL, MA, and RU samples from the chemical-cure group compared with that from the dual-cure group. On the contrary, no significant differences were observed in the GL and PV wear simulation specimens that were cured using the two different curing modes. These findings may be attributed to the different compositions of the materials used. Polymerization of dual-cure resin luting cements can be activated either by inducing a photo-initiator (e.g., camphorquinone) or by breaking the molecules of a chemical-initiator (e.g., benzoyl peroxide) such that free radicals are formed to initiate the polymerization reactions (37,38). The contents of the initiators for photo- and chemical curing in resin luting cements differ depending on the material (39). For instance, the benzoyl peroxide content in some types of resin luting cements is twice that of other materials. Some resin luting cements are overly dependent on photo-curing, which may lead to inadequate polymerization and performance in clinical situations

(40). Unfortunately, detailed information from manufacturers about the contents of photo and chemical initiators in resin luting cement is very limited; therefore, further comparisons are difficult.

On the basis of the results of the present study, the null hypothesis that the simulated localized wear of resin luting cements for universal adhesive systems would not be influenced by the type of material or the curing mode used is partially rejected.

The results of the present study indicate that the simulated localized wear of resin luting cements for universal adhesive systems is material dependent. In addition, in most cases, dual curing appears to result in greater wear resistance of the resin luting cement than chemical curing; however, some resin luting cements showed no significant differences between the two curing modes. Therefore, the influence of the curing mode on resin luting cements seems to be material dependent. In clinical situations with restricted light exposure conditions, care should be taken when choosing a resin luting cement for universal adhesive systems that is effectively cured in the chemical-cure mode.

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## Conflict of interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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