

Mechanical Properties and Sliding-impact Wear Resistance of Self-adhesive Resin Cements

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Clinical Relevance

Mechanical properties and wear resistance are important parameters for the selection of a resin luting cement. For wear resistance, the self-adhesive resin cements generally show significantly lower values than the conventional resin cements.

SUMMARY

The present study determined the mechanical properties and impact-sliding wear characteristics of self-adhesive resin cements. Five self-adhesive resin cements were used: G-CEM

LinkAce, BeautiCem SA, Maxcem Elite, Clearfil SA Automix, and RelyX Unicem 2. Clearfil Esthetic Cement was employed as a control material. Six specimens for each resin cement were used to determine flexural strength, elastic modulus, and resilience according to ISO specification #4049. Ten specimens for each resin cement were used to determine the wear characteristics using an impact-sliding wear testing apparatus. Wear was generated using a stainless-steel ball bearing mounted inside a collet assembly. The maximum facet depth and volume loss were determined using a noncontact profilometer in combination with confocal laser scanning microscopy. Data were evaluated using analysis of variance followed by the Tukey honestly significantly different test ($\alpha=0.05$). The flexural strength of the resin cements ranged from 68.4 to 144.2 MPa; the elastic modulus ranged from 4.4 to 10.6 GPa; and the resilience ranged from 4.5 to 12.0 MJ/m³. The results for the maximum facet depth ranged from 25.2 to 235.9 μ m, and volume loss ranged from 0.0107 to 0.5258 mm³. The flexural properties and wear resistance were found to vary depending upon the self-adhesive resin

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cement tested. The self-adhesive cements tended to have lower mechanical properties than the conventional resin cement. All self-adhesive resin cements, apart from G-CEM LinkAce, demonstrated significantly poorer wear resistance than did the conventional resin cement.

INTRODUCTION

Self-adhesive resin cements utilized as luting agents are defined as cements based on filled polymers that adhere to the tooth structure in the absence of tooth surface pretreatment.^{1,2} Therefore, self-adhesive resin cements are considered easier to apply compared with conventional resin cements.³ The application procedure is simple, and no postoperative sensitivity is expected because the smear layer is not removed during the cementation process. These cements contain acidic functional monomers that demineralize the tooth structure and promote infiltration of the resin components into the etched tooth substrate.^{1,2} In addition, the setting reactions of most self-adhesive resin cements use a dual curing process based on the incorporation of photo-initiators along with redox initiators. However, in contrast to their simplified application, the compositions of self-adhesive resin cements are complicated. Moreover, incompatibility between acidic functional monomers and other resinous components can have adverse effects on the mechanical properties of self-adhesive resin cements. Nevertheless, previous studies^{4,5} have demonstrated that self-adhesive resin cements are mechanically stronger than zinc phosphate, zinc polycarboxylate, glass ionomer, and resin-modified glass ionomer cements when comparing their flexural and compressive strengths. In addition, it has been established that self-adhesive resin cements possess similar flexural strengths to those of conventional resin cements.⁶⁻⁸

From the standpoint of clinical conditions, one of the challenges for luting cements is marginal integrity. To select suitable luting cements for specific clinical conditions, it is important to evaluate the wear performance characteristics of the cement to achieve long-lasting restorations. Marginal gap formation may lead to a variety of concerns, including marginal staining, secondary caries, bonding failure, restoration fracture, and postoperative sensitivity.⁹ In addition, dental clinicians require more information related to the clinical relevance of the mechanical properties of more recently developed self-adhesive resin cements, including detail about their wear resistance.

Several studies^{10,12} have evaluated marginal gaps in an effort to assess the potential for cement loss at the margins of restorations and to yield more information concerning the expected quality of restorations. However, little information exists concerning the wear resistance properties of recently developed self-adhesive resin cements. The purpose of the present study was to assess the flexural properties and wear properties of self-adhesive resin cements and to compare these properties with those of a conventional resin cement. The null hypothesis to be tested was that there are no significant differences in wear and mechanical properties between the conventional resin cement and the self-adhesive resin cements.

METHODS AND MATERIALS

Materials Used

Five self-adhesive resin cements were tested: G-CEM LinkAce (GL; GC Corp., Tokyo, Japan); BeautiCem SA Auto-mixing (BC; Shofu Inc, Kyoto, Japan); Maxcem Elite (ME; Kerr Corp, Orange, CA, USA); Clearfil SA Luting Automix (SA; Kuraray Noritake Dental Inc, Tokyo, Japan); and RelyX Unicem 2 (RU; 3M ESPE Dental Products, St Paul, MN, USA). A conventional resin cement, Clearfil Esthetic Cement (EC; Kuraray Noritake Dental Inc), was employed as a control material. The test materials and their components are listed in Table 1. An Optilux 501 visible-light-curing unit (sds Kerr, Danbury, CT, USA) was employed, and the light irradiance (average 800 mW/cm²) of the curing unit was checked using a dental radiometer (Model 100, Kerr).

Inorganic Filler Content

The inorganic filler content of the materials was measured using thermogravimetry/differential thermal analysis employing a 6300 thermogravimeter (Seiko Instruments, Tokyo, Japan). For each resin cement tested, a mixed cement paste sample (approximately 50 mg) was heated in the thermogravimeter from 25°C to 800°C at a heating rate of 10°C/min until the organic components were completely consumed. The weight of the residual cement paste was measured and the inorganic filler content (wt%) was calculated. Three measurements were conducted to obtain an average inorganic filler content (wt%).

Coefficient of Linear Thermal Expansion

The coefficients of linear thermal expansion of the test materials were measured using a thermome-

Table 1: Primary Characteristics of the Six Cement Materials Studied

Self-adhesive Resin Cements	Shade	Manufacturer	Main Components ^a	Code
G-CEM LinkAce Lot No. 1402271	A2	GC Corp, Tokyo, Japan	UDMA, dimethacrylate, phosphonate monomer, γ -methacryloxypropyltrimethoxysilane, α , α -dimethylbenzyl hydroperoxide, fluoro alumino silicate glass, silicon dioxide, initiator, inhibitor, pigment	GL
BeautiCem SA Lot No. 081346	Ivory	Shofu Inc, Kyoto, Japan	UDMA, HEMA, carboxylic acid monomer, phosphonate monomer, fluoro alumino silicate glass, zirconium silicate filler (amorphous), polymerization initiator, others	BC
Maxcem Elite Lot No. 4947576	Brown	Kerr Corp, Orange, CA, USA	Bis-GMA, UDMA, GPDM, glyceroldimethacrylate, mono-, di-, and multi-methacrylate co-monomers, CQ, barium alumino borosilicate glass, fluoro alumino silicate glass, stabilizer, others	ME
Clearfil SA Automix Lot No. 6L0011	A2	Kuraray Noritake Dental Inc, Tokyo, Japan	Bis-GMA, TEGDMA, MDP, hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, silanated barium glass filler, silanated colloidal silica, surface-treated sodium fluoride, CQ, initiator, benzoyl peroxide, accelerators, pigments	SA
RelyX Unicem 2 Lot No. 6L0011	A2	3M ESPE Dental Products, St Paul, MN, USA	Propanediyl dimethacrylate and phosphorus oxide, substitute dimethacrylate, TEDGMA, 2-propenoic acid, 2-methyl-, 1,1'-[1-(hydroxymethyl)-1,2-ethanediyl]ester, silane-treated glass powder, silane-treated silica, sodium persulfate, glass powder, <i>tert</i> -butyl peroxy-3,5,5-trimethylhexanoate, copper (II) acetate monohydrate, 1,12-dodecane dimethacrylate, 1-benzyl-5-phenyl-barbic-acid, calcium salt, sodium <i>p</i> -toluenesulfonate, calcium hydroxide, methacrylated aliphatic amine, titanium dioxide	RU
Esthetic Cement Lot No. 0037AA	A2	Kuraray Noritake Dental Inc, Tokyo, Japan	Bis-GMA, TEGDMA, hydrophobic aromatic dimethacrylate, hydrophilic aliphatic dimethacrylate, silanated barium glass filler, silanated silica filler, colloidal silica, benzoyl peroxide, accelerator, CQ, initiators, pigments, others	EC
Abbreviations: Bis-GMA, 2,2-bis[4-(2-hydroxy-3-methacryloyloxypropoxy)phenyl]propane; CQ, <i>DL</i> -camphorquinone; GPDM, glyceroldimethacrylate dihydrogen phosphate; HEMA, 2-hydroxyethyl methacrylate; MDP, 10-methacryloyloxydecyl dihydrogen phosphate; TEGDMA, triethyleneglycol dimethacrylate; UDMA, urethane dimethacrylate. Indicate no statistically significant differences among its members (Tukey honestly significantly different [HSD] test, $p > 0.05$). ^a According to each manufacturer's Material Safety Data Sheet.				

chanical analyzer (TMA/SS 6300, Seiko Instruments). For each resin cement tested, the mixed paste was compacted into a stainless-steel split mold of dimensions 25 mm \times 2 mm \times 2 mm, which was positioned on a glass slide. For resin cement curing within the mold, the central 5-mm section, followed by the adjacent sections, was irradiated for 30 seconds, and, finally, each end was irradiated in turn for the same period. The specimens were stored under dark conditions at 25°C for 24 hours and subsequently fixed in a vice and cut in half (for a length of 12.5 mm) with a diamond saw before the measurements were conducted. Three specimens for each material were prepared and separately tested in the thermomechanical analyzer at a heating rate of 2°C/min from 25°C to 130°C. The average coefficient of linear thermal expansion ($\times 10^{-6}/^{\circ}\text{C}$),

over a temperature range 30°C to 80°C, was thereby obtained.

Flexural Strength and Elastic Modulus Measurement

The flexural properties were tested according to ISO specification #4049. The test samples were obtained following an equivalent procedure to that described above for evaluation of the coefficients of linear thermal expansion, except that they were not cut in half. After the removal of the hardened specimen from the mold, all six surfaces were wet ground with #1200 silicon carbide (SiC) papers (Fuji Star Type DDC, Sankyo Rikagaku Co, Saitama, Japan). The specimens were then stored for 24 hours in distilled water at 37°C prior to testing. Six specimens for each of the six resin cements were

subjected to a three-point bending flexural strength test using a universal testing instrument (Type 5500R, Instron Corp, Canton, MA, USA) with a span length of 20.0 mm at a cross-head speed of 1.0 mm/min until the specimen fractured. The specimens were kept moist during testing. The peak breaking stress, modulus of elasticity, and resilience were determined from the stress-strain curve of each sample using the Bluehill Ver 2.5 computer software (Instron Corp) linked to the testing instrument.

Impact-sliding Wear Simulation

Ten specimens for each of the six resin cements were tested to determine the wear behavior using the impact-sliding wear testing apparatus (K655-05, Tokyo Giken Inc, Tokyo, Japan). For each specimen, the mixed resin cement paste was placed into a cylindrical Teflon mold (6 mm in diameter, 2 mm in height). The cement paste was cured for 30 seconds and stored under dark conditions for 24 hours in distilled water at 37°C. The flat surfaces of each specimen were polished to 4000 grit using a graded sequence of SiC papers. Specimens were attached with a small amount of model-repair glue (Zapit, Dental Ventures of America Inc, Corona, CA, USA) to the centers of custom fixtures fabricated from a cold-cure acrylic resin (Tray Resin II, Shofu Inc).

The antagonist for the impact-sliding wear simulation was a stainless-steel ball bearing of radius $r = 2.387$ mm that was mounted inside a collet assembly. The simulator incorporated a plastic water bath that provided water at a temperature of 37°C constantly, and four of the custom wear fixtures were mounted inside the bath. During the course of the wear simulation test, the antagonists directly impacted the specimens from above with a maximum force of 50 N at a rate of 0.5 Hz, and the antagonists subsequently slid horizontally for a distance of 2 mm. Each specimen was subjected to 50,000 cycles of the impact-sliding motion.

Wear Measurements

Following the wear simulation test, the specimens were ultrasonically cleaned in distilled water for three minutes and then profiled using a confocal laser scanning microscope (VK-9710, Keyence, Osaka, Japan) with built-in analysis software (VK analyzer, Keyence). The maximum depth (μm) and volume loss (mm^3) of the wear facets were thereby determined.

Scanning Electron Microscopy Observations

The surfaces of the cured resin cement samples were polished to a high gloss with abrasive discs (Fuji Star Type DDC, Sankyo Rikagaku Co) followed by a series of diamond pastes down to a 0.25- μm particle size (DP-Paste, Struers, Ballerup, Denmark). The polished surfaces were then subjected to unfiltered argon-ion beam etching (IIS-200ER, Elionix, Tokyo, Japan) for 40 seconds perpendicular to the polished surface at an accelerating voltage of 1.0 kV and ion current density of 0.4 mA/cm^2 . The surfaces were then coated in a vacuum evaporator (Quick Coater Type SC-701; Sanyu Denshi Inc, Tokyo, Japan) with a thin film of gold. Examinations of the surfaces were conducted by scanning electron microscopy (SEM) using an Elionix FE-8000 instrument (Elionix, Tokyo, Japan) with an operating voltage of 10 kV and a magnification of 5000 \times .

SEM examinations were conducted on the wear facets of the six resin cement samples after impact-sliding wear simulation. Representative samples for each resin cement were sputter-coated with gold. Examinations of the coated surfaces were conducted using the SEM with an operating voltage of 10 kV and magnifications of 40 \times and 2500 \times .

Statistical Analysis

The data for each material were subjected to analysis of variance followed by the Tukey honestly significantly difference (HSD) test at a significance level of 0.05. The statistical analysis was conducted using the Sigma Plot software system (Ver. 11.0; SPSS Inc, Chicago, IL, USA).

RESULTS

Inorganic Filler Content and Coefficient of Thermal Expansion

The average inorganic filler contents and coefficients of thermal expansion are listed in Table 2. The average inorganic filler contents of the resin cements ranged from 55.3 to 67.9 wt%. The highest inorganic filler content was determined for EC, whereas GL showed the lowest value of all the test materials, and these differences were statistically significant. The average coefficients of thermal expansion of the resin cements ranged from 37.7 to 51.6 $\times 10^{-6}/^\circ\text{C}$. A significantly low value was exhibited by RU, whereas ME showed a significantly higher value than all other tested resin cements.

Table 2: Inorganic Filler Contents and Thermal Expansion Coefficients of the Cement Materials Studied

Code	Inorganic Filler Content, wt% ^a	Tukey Group ^b	Thermal Expansion Coefficient, $\times 10^{-6}/^{\circ}\text{C}$ (from 30°C to 80°C) ^a	Tukey Group ^b
GL	55.3 (0.2)	F	44.7 (1.6)	B
BC	59.6 (0.2)	E	42.8 (1.5)	B
ME	66.9 (0.2)	B	51.6 (0.8)	A
SA	65.8 (0.2)	C	45.3 (0.9)	B
RU	63.6 (0.2)	D	37.7 (0.2)	C
EC	67.9 (0.3)	A	41.8 (2.0)	B

Abbreviations: BC, BeautiCem SA; EC, Esthetic Cement; GL, G-CEM LinkAce G-CEM LinkAce; ME, Maxcem Elite; RU, RelyX Unicem 2; SA, Clearfil SA Automix.
^a Mean (standard deviation), $n = 6$.
^b Equivalent group letters indicate no statistically significant differences among its members (Tukey honestly significantly different [HSD] test, $p > 0.05$).

Flexural Properties

The results of flexural property testing are listed in Table 3. The average flexural strength of the resin cements ranged from 68.4 to 144.2 MPa, and the average elastic modulus ranged from 4.4 to 10.6 GPa. Tukey HSD test indicated that BC exhibited the highest flexural strength of all test resin cements, and no significant difference was obtained when BC was compared to the control material EC. Regarding the elastic modulus, EC demonstrated the highest value. Self-adhesive resin cements BC, GL, and RU exhibited higher values compared to all other self-adhesive resin cements, and no significant differences were obtained among them. The average resilience ranged from 4.5 to 12.0 MJ/m³. The control material EC exhibited the highest resilience.

Impact-sliding Wear

The average wear values (maximum facet depth and volume loss) for the six resin cements are summa-

Table 4: Maximum Facet Depth and Volume Loss of the Cement Materials Studied

Code	Maximum Facet Depth, μm ^a	Tukey Group ^b	Volume Loss, mm ^{3a}	Tukey Group ^b
GL	28.6 (1.5)	E	0.0110 (0.003)	D
BC	122.3 (31.7)	D	0.1712 (0.059)	C
ME	239.5 (13.9)	A	0.5258 (0.051)	A
SA	187.5 (30.8)	C	0.3075 (0.103)	B
RU	197.8 (44.9)	B	0.2060 (0.046)	C
EC	25.2 (10.2)	E	0.0107 (0.005)	D

Abbreviations: BC, BeautiCem SA; EC, Esthetic Cement; GL, G-CEM LinkAce G-CEM LinkAce; ME, Maxcem Elite; RU, RelyX Unicem 2; SA, Clearfil SA Automix.
^a Mean (standard deviation), $n = 6$.
^b Equivalent group letters indicate no statistically significant differences among its members (Tukey honestly significantly different [HSD] test, $p > 0.05$).

rized in Table 4. The rank orders of all six resin cements were similar with regard to both facet wear depth and volumetric loss. Cements EC and GL demonstrated significantly lower maximum facet depth and volume loss when compared to all other resin cements. In contrast, ME exhibited the highest maximum facet depth and volume loss when compared to all other resin cements, and all differences were statistically significant.

SEM Observations

SEM examinations of the six resin cements after argon-ion etching are presented in Figure 1a-f. Differences in filler particle size, shape, and distribution are clearly observable. For GL, RU, and ME, 0.5-5- μm irregularly shaped glass filler particles are observed, as shown in Figure 1a, c, and e, respectively. For BC, 0.2-5 μm -sized spherical silica filler particles and 0.2-1 μm irregular glass filler particles are observed in Figure 1b. SA and EC include relatively large irregular glass filler particles mea-

Table 3: Flexural Strength, Elastic Modulus, and Resilience of the Cement Materials Studied

Code	Flexural Strength, MPa ^a	Tukey Group ^b	Flexural Modulus, GPa ^a	Tukey Group ^b	Resilience, MJ/mm ^{3a}	Tukey Group ^b
GL	121.4 (7.9)	B	7.2 (0.4)	B	9.5 (2.1)	B
BC	144.2 (12.2)	A	7.7 (0.5)	B	8.5 (2.0)	B,C
ME	68.4 (3.9)	D	4.4 (0.3)	D	4.5 (0.5)	D
SA	86.6 (8.5)	C	5.3 (0.5)	C	6.3 (1.5)	C,D
RU	99.7 (8.6)	C	7.2 (0.4)	B	6.0 (0.9)	C,D
EC	138.5 (3.8)	A	10.6 (0.8)	A	12.0 (2.0)	A

Abbreviations: BC, BeautiCem SA; EC, Esthetic Cement; GL, G-CEM LinkAce G-CEM LinkAce; ME, Maxcem Elite; RU, RelyX Unicem 2; SA, Clearfil SA Automix.
^a Mean (standard deviation), $n = 6$.
^b Equivalent group letters indicate no statistically significant differences among its members (Tukey honestly significantly different [HSD] test, $p > 0.05$).

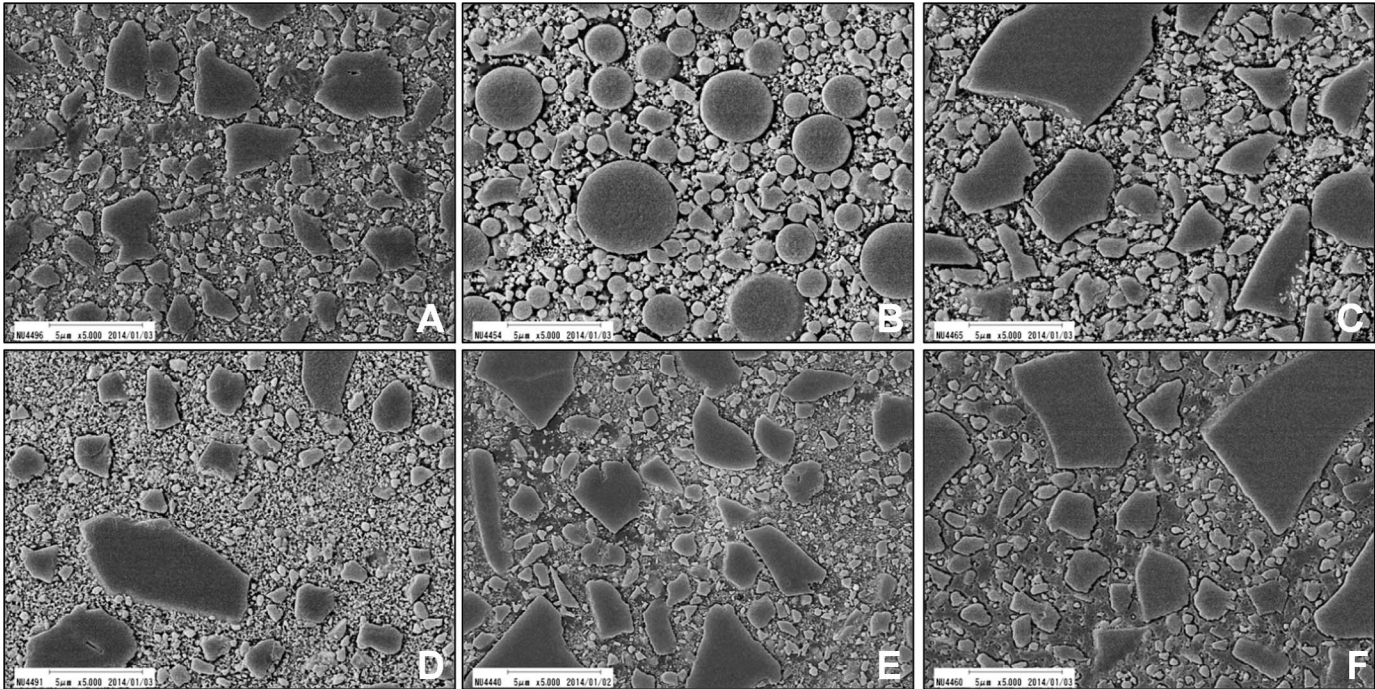


Figure 1. SEM micrographs at a 5000 \times magnification of the argon-ion etched surfaces of the six cement materials studied, as follows. (A) G-CEM LinkAce argon-ion etched surface at 5000 \times . (B) BeautiCem SA argon-ion etched surface at 5000 \times . (C) Maxcem Elite argon-ion etched surface at 5000 \times . (D) Clearfil SA Automix argon-ion etched surface at 5000 \times . (E) RelyX Unicem 2 argon-ion etched surface at 5000 \times . (F) Esthetic Cement argon-ion etched surface at 5000 \times .

suring approximately 0.2-10 μm in size, as shown in Figure 1d and f.

Representative SEM images of wear facets after impact-sliding wear testing are presented in Figure 2a-h. For GL and EC, the SEM examinations given

in Figure 2a and g, respectively, reveal relatively flat and smooth surfaces, although some fine cracks are observed on the worn surfaces, as shown in Figures 2b and h for GL and EC, respectively. However, all other self-adhesive resin cements exhibited rougher

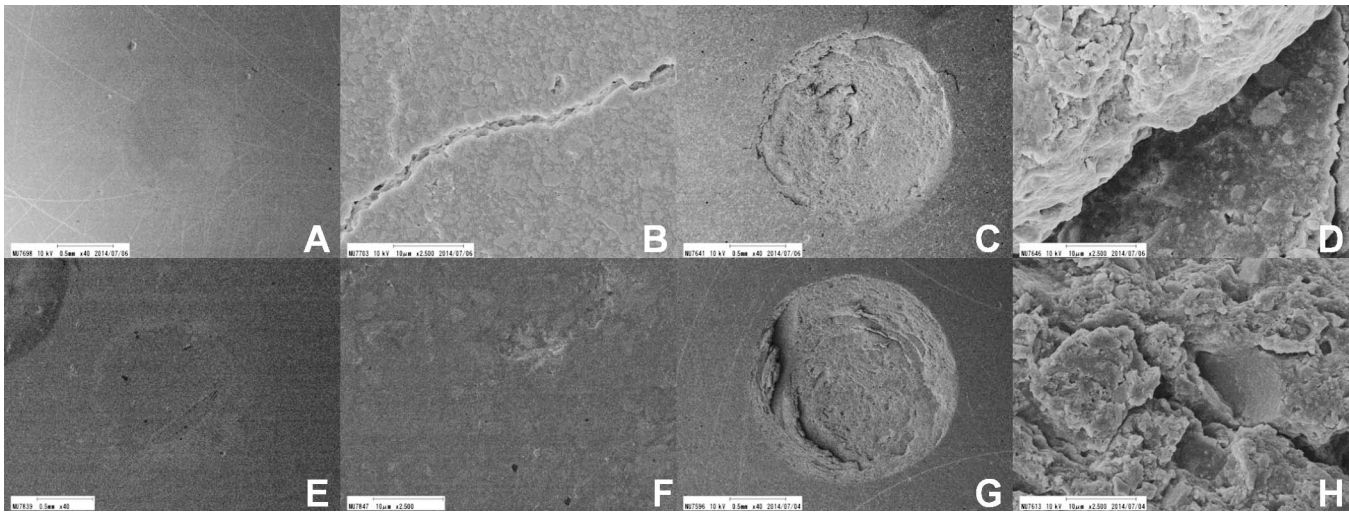


Figure 2. SEM micrographs at 40 \times and 2500 \times magnifications of the surfaces of representative samples of the six cement materials subjected to impact-sliding wear testing. (A) G-CEM LinkAce Impact-sliding wear facet at 40 \times . (B) G-CEM LinkAce Impact-sliding wear near center of facet at 2500 \times . (C) Clearfil SA Automix Impact-sliding wear facet 40 \times . (D) Clearfil SA Automix Impact-sliding wear near center of facet at 2500 \times . (E) RelyX Unicem 2 Impact-sliding wear facet 40 \times . (F) RelyX Unicem 2 Impact-sliding wear near center of facet at 2500 \times . (G) Esthetic Cement Impact-sliding wear facet 40 \times . (H) Esthetic Cement Impact-sliding wear near center of facet at 2500 \times .

and deeper facets than those of either GL or the control material EC. Similar wear patterns are observed for the more highly worn self-adhesive resin cement samples, with some plucking of filler particles from the surface and obvious cracks, as shown in Figure 2c-f.

DISCUSSION

Improvements in the resin matrix and filler components of direct composite restorations have resulted in markedly improved materials for applications in the posterior region.^{13,14} However, dental professionals remain concerned about the mechanical properties and wear resistance of these materials for application to larger posterior lesions. Indirect restorations have been selected, rather than direct restorations, as the standard procedure for the posterior region because of their durable properties, ease of fabrication in a precise anatomical form, and adaptation to adjacent teeth.¹⁵ At present, metal alloys, resin composites, and ceramics are used as indirect restorative materials. However, to achieve durable bonds between an indirect restoration and the surrounding tooth structure, each of these materials requires several pretreatment steps for luting.

Therefore, simplified self-adhesive resin cements for luting of restoratives have been developed that can reduce the clinical steps required for the luting procedure, mitigate technique sensitivity, and diminish postoperative sensitivity.^{16,17} In addition, to avoid an insufficient degree of conversion when light delivery from a curing unit is inhibited as a result of obstructions by overlying indirect restorations, self-adhesive resin cements have been developed that incorporate a dual-polymerization system. This type of cement contains inorganic fillers, matrix resins, initiator systems, and acidic functional monomers. However, a major concern has been expressed regarding a reduced degree of conversion due to the presence of acidic functional monomers because of the risk of lower pH^{18,19} and interference with the reaction between the tertiary amine and camphorquinone.²⁰⁻²² Therefore, to predict *in vivo* performance of self-adhesive resin cements, it is important to determine their endurance characteristics by evaluating their mechanical properties and wear resistance.

The results for inorganic filler content and thermal expansion of the resin cements appeared to be material dependent. Regarding the filler content, it is considered that increasing the amount of inorganic filler content plays an important role in

enhancing the mechanical properties, reducing the polymerization shrinkage, and increasing the wear resistance. Although a high filler content is desirable for resin-based materials, it is difficult to include a large amount of filler because of the need to maintain an appropriate viscosity for luting and to ensure a suitable film thickness.

Thermal expansion is an important property of luting cements when considering the longevity of restorations. Temperature cycling may induce dimensional changes in the materials and rupture of the bond interface not only between the luting cement and the tooth structure, but also between the luting cement and the restoration as a result of differences in the thermal expansion properties of the individual materials. Because the thermal expansion of the restorative material and luting cements usually does not match that of the tooth structure, a differential expansion occurs that may result in leakage of oral fluids into the interface between the restoration and the tooth. From the results of this study, thermal expansion of the six resin cements ranged from 37.7 to 51.6 $\times 10^{-6}$ m/°C. In addition, in spite of the higher inorganic filler content, ME exhibited a significantly higher average thermal expansion coefficient than all other resin cements. A higher inorganic filler content has been considered to reduce thermal expansion, but the results obtained in this study suggested that other factors, such as the type of matrix resin and size of the filler, might have a significant influence on the thermal properties. Considering the long-term durability of indirect restorations, further improvement of thermal properties is needed in the resin cements.

Fracture-related material properties, such as fracture resistance, elasticity, and the marginal degradation of materials under stress, have typically been evaluated by determining material parameters such as flexural strength, flexural modulus, and fracture toughness.^{23,24} Although flexural strength under constant loading may not reflect intraoral conditions, these values are helpful in comparing materials under controlled conditions.²⁵ From the results of flexural testing, the control material EC tended to have a higher flexural strength than the self-adhesive cements considered. However, BS and GL demonstrated a flexural strength similar to that of EC. A previous study revealed a correlation between the filler content, filler size, and the distribution of filler particles in resin-based materials and their material properties.^{26,27} However, analysis of those results in this study showed no significant correlation, so other factors appear to be

more important. In addition, it is speculated that the functional monomers contained in self-adhesive cements might affect the degree of conversion of the cement itself.

It has been suggested that the desirable modulus of elasticity for a luting cement lies between those of the restorations and mineralized tooth structure, which contributes to a reduction of interfacial stress concentrations. Furthermore, resistance to plastic deformation may increase the resistance to marginal gap formation and cement fracture, and therefore a high proportional limit and resilience are preferred.²⁸ For resilience, the self-adhesive cements tended to exhibit lower values than that of the control material EC. Thus, the lower elastic modulus and resilience of self-adhesive cements may remain a disadvantage in preventing deformation and fracture of brittle restorations, such as all-ceramic and machinery-milled restorations.

Many types of filler systems, monomer systems, and coupling agents have been developed to improve the mechanical properties and wear resistance of resin composites. Resin luting cements, including self-adhesive resin cements, have been developed using resin composite technology. Thus, these types of materials might have similar characteristics to those of resin composites, and the wear resistance of the cement itself should be evaluated. Wear progression of luting cements at the margin may lead to gaps between a restoration and the surrounding tooth structure and may create a vulnerable region that serves as a foothold for crack propagation in the cement as a result of mastication. Increasing gap formation may induce secondary caries, bonding failure, restorative fracture in the body or at the margins, and postoperative sensitivity.⁹

Clinical criteria have been used to assess marginal integrity in long-term clinical trials.^{29,30} However, these investigations are costly, time-intensive, and often technically challenging to complete. In contrast, simulated wear measurement could provide a rapid means with which to examine relative wear rates among materials and to predict expected clinical performance.^{31,32}

In the present study, a sliding-impact wear apparatus was used as a two-body contact wear simulator. In a three-body contact wear model, abrasion, adhesive, and erosion wear are likely the dominant forces, but their relative contributions are not known. However, for a two-body contact wear model, it is considered that abrasion, adhesive wear, and fatigue or attrition are dominant.³³ Although

three-body wear testing with an abrasive medium has been used frequently to simulate the mastication of food, the use of abrasive media influences the results as a result of the formation of an embedded layer of abrasive medium and creates a difficulty with standardizing these mixtures or maintaining a consistent viscosity and composition during the entire wear simulation process.³⁴ These considerations suggest that the two-body wear test employed in the present study has the advantages of eliminating variability and establishing a simplified model.

The wear values (maximum facet depth and volume loss) for the six resin cements appear to be material dependent, as was also determined for the mechanical properties. The self-adhesive cements, apart from GL, demonstrated significantly lower wear resistance than did the control material EC. Therefore, the null hypothesis that self-adhesive resin cements will show significant differences in wear resistance and mechanical properties from the control resin cement was not completely rejected. From the SEM images of wear facets after impact-sliding wear testing, GL and EC revealed relatively flat and smooth surfaces, in addition to some fine cracks on the worn surfaces. On the other hand, the other self-adhesive cements exhibited rougher and deeper facets than did both GL and EC. The other materials also exhibited a similar wear pattern, with some plucking of filler particles from the surface. Considering the impact and sliding force administered by the wear equipment employed, it could be considered that smaller filler particles would preserve the surface texture even if some filler were plucked from the resin matrix and might inhibit crack propagation.¹⁰

It has been reported^{35,36} that improvement in the degree of conversion of resin composites is expected to increase wear resistance and mechanical properties. In addition, previous studies suggested that dual-cured self-adhesive resin cements with photo activation would obtain a superior degree of conversion compared with self-curing alone, because acidic functional monomers act negatively on the self-cure setting reaction by their relatively lower pH value^{18,19} and interfere with the reaction between the tertiary amine and camphorquinone.²⁰⁻²² Although the specimens for mechanical properties and wear resistance were evaluated after photo activation, it is possible that the acidic functional monomers influenced the polymerization of the self-adhesive resin cements.

While mechanical properties and wear behavior are not the only parameters for consideration in the

selection of resin cements, these characteristics can provide valuable information to dental professionals.

CONCLUSIONS

The results of this study showed that the self-adhesive cements tended to have poorer mechanical properties than the conventional resin cements. For wear resistance, the self-adhesive resin cements, apart from GL, showed significantly lower values than did the conventional resin cements. These results augment the information base available to the profession and provide guidance for clinicians in the selection of resin cements.

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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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REFERENCES

1. Radovic I, Monticelli F, Goracci C, Vulicevic ZR, & Ferrari M (2008) Self-adhesive resin cements: A literature review *Journal of Adhesive Dentistry* **10**(4) 251-258.
2. Ferracane JL, Stansbury JW, & Burke FJT (2011) Review article self-adhesive resin cements—Chemistry, properties and clinical considerations *Journal of Oral Rehabilitation* **38**(4) 295-314.
3. Burke FJT, & Birmingham RJC (2006) A practice-based evaluation of the handling of new self-adhesive universal resin luting material *International Dental Journal* **56**(3) 142-146.
4. Piwowarczyk A, & Lauer H-C (2003) Mechanical properties of luting cements after water storage *Operative Dentistry* **28**(5) 535-542.
5. Kumbuloglu O, Lassila LV, User A, & Vallittu PK (2004) A study of the physical and chemical properties of four resin composite luting cements *International Journal of Prosthodontics* **17**(3) 357-363.
6. Saskalauskaite E, Tam LE, & McComb D (2006) Flexural strength, elastic modulus, and pH profile of self-etch resin luting cements *Journal of Prosthodontics* **17**(4) 262-268.
7. Behr M, Rosentritt M, Loher H, Kolbeck C, Templer C, Stemplinger B, Kopzon V, & Handel G (2008) Changes of cement properties caused by mixing errors: The therapeutic range of different cement types *Dental Materials* **24**(9) 1187-1193.
8. Nakamura T, Wakabayashi K, Kinuta S, Nishida H, Miyamae M, & Yatani H (2010) Mechanical properties of new self-adhesive resin-based cement *Journal of Prosthodontic Research* **54**(2) 59-64.
9. Ibarra G, Johnson GH, Geurtsen W, & Vargas MA (2007) Microleakage of porcelain veneer restorations bonded to enamel and dentin with a new self-adhesive resin-based dental cement *Dental Materials* **23**(2) 218-225.
10. Shinkai K, Suzuki S, Leinfelder KF, & Katoh Y (1995) Effect of gap dimension on wear resistance of luting cements *American Journal of Dentistry* **8**(3) 149-151.
11. Braga RR, Condon JR, & Ferracane JL (2002) In vitro wear simulation measurements of composite versus resin-modified glass ionomer luting cements for all-ceramic restorations *Journal of Esthetic and Restorative Dentistry* **14**(6) 368-376.
12. Belli R, Pelka M, Petschelt A, & Lohbauer U (2009) In vitro wear gap formation of self-adhesive resin cements: A CLSM evaluation *Journal of Dentistry* **37**(12) 984-993.
13. Lutz F, Phillips RW, Roulet JF, & Setcos JC (1984) In vivo and in vitro wear of potential posterior composites *Journal of Dental Research* **63**(6) 914-920.
14. Heintze SD, Barkmeier WW, Latta MA, & Rousson V (2011) Round robin test: Wear of nine restorative materials in six different wear simulators—Supplement to the round robin test of 2005 *Dental Materials* **27**(2) e1-e9.
15. Wassell RW, Walls AW, & McCabe JF (1995) Direct composite inlays versus conventional composite restorations: Three year clinical results *British Dental Journal* **179**(9) 343-349.
16. Monticelli F, Osorio R, Mazzitelli C, Ferrari M, & Toledano M (2008) Limited decalcification/diffusion of self-adhesive cements into dentin *Journal of Dental Research* **87**(10) 974-979.
17. Cantoro A, Goracci C, Carvalho CA, Coniglio I, & Ferrari M (2009) Bonding potential of self-adhesive luting agents used at different temperatures to lute composite onlay *Journal of Dentistry* **37**(6) 454-461.
18. Vrochari AD, Eliades G, Hellwig E, & Wrbas KT (2009) Curing efficiency of four self-etching, self-adhesive resin cements *Dental Materials* **25**(9) 1104-1108.
19. Zorzin J, Petschelt A, Ebert J, & Lohbauer U (2012) pH neutralization and influence on mechanical strength in self-adhesive resin luting agents *Dental Materials* **28**(6) 672-679.
20. Sanares AME, Itthagarun A, King NM, Tay FR, & Pashley DH (2001) Adverse surface interactions between one-bottle light-cured adhesives and chemical-cured composites *Dental Materials* **17**(6) 542-556.
21. Suh BI, Feng L, Pashley DH, & Tay FR (2003) Factors contributing to the incompatibility between simplified-step adhesives and chemically-cured or dual-cured composites. Part III. Effect of acidic resin monomers *Journal of Adhesive Dentistry* **5**(4) 267-282.

22. Salz U, Zimmermann J, & Salzer T (2005) Self-curing, self-adhesive cements systems *Journal of Adhesive Dentistry* **7**(1) 7-17.
23. Watanabe H, Khera SC, Vargas MA, & Qian F (2008) Fracture toughness comparison of six resin composites *Dental Materials* **24**(3) 418-425.
24. Sumino N, Tsubota K, Takamizawa T, Shiratsuchi K, Miyazaki M, & Latta MA (2013) Comparison of the wear and flexural characteristics of flowable resin composites for posterior lesions *Acta Odontologica Scandinavica* **71**(3-4) 820-827.
25. Lang R, Rosentritt M, Behr M, & Handel G (2003) Fracture resistance of PMMA and resin matrix composite-based interim FPD materials *International Journal of Prosthodontics* **16**(4) 381-384.
26. Jain P, & Belcher M (2000) Microleakage of Class II resin-based composite restorations with flowable composite in the proximal box *American Journal of Dentistry* **13**(5) 235-238.
27. Turssi CP, Ferracane JL, & Ferracane LL (2006) Wear and fatigue behavior of nano-structured dental resin composites *Journal of Biomedical Materials Research Part B: Applied Biomaterials* **78**(1) 196-203.
28. Li ZC, & White SN (1999) Mechanical properties of dental luting cements *Journal of Prosthetic Dentistry* **81**(5) 597-609.
29. Frankenberger R, Reinelt C, Petschelt A, & Kramer N (2009) Operator vs. material influence on clinical outcome of bonded ceramic inlays *Dental Materials* **25**(8) 960-968.
30. Peumans M, Voet MJ, De Munk J, Van Landuyt K, Van Ende B, & Van Meerbeek B (2013) Four-year clinical evaluation of a self-adhesive luting agent for ceramic inlays *Clinical Oral Investigations* **17**(3) 739-750.
31. Barkmeier WW, Erickson RL, Latta MA, & Wilwerding TM (2008) Wear simulation of resin composites and the relationship to clinical wear *Operative Dentistry* **33**(2) 177-182.
32. Barkmeier WW, Erickson RL, Latta MA, & Wilwerding TM (2013) Wear rates of resin composites *Operative Dentistry* **38**(2) 226-233.
33. Condon JR, & Ferracane JL (1997) Factors effecting dental composite wear in vitro *Journal of Biomedical Material Research Part A* **38**(4) 303-313.
34. Heintze SD (2006) How to qualify and validate wear simulation devices and methods *Dental Materials* **22**(8) 712-734.
35. Ferracane JL, & Berge HX (1995) Fracture toughness of experimental dental composites aged in ethanol *Journal of Dental Research* **74**(7) 1418-1423.
36. Ferracane JL, Mitchem JC, Condon JR, & Todd T (1997) Wear and marginal breakdown of composites with various degree of cure *Journal of Dental Research* **76**(8) 1508-1516.