# Simulated Wear of Self-Adhesive Resin Cements

T Takamizawa • WW Barkmeier • MA Latta TP Berry • A Tsujimoto • M Miyazaki

#### **Clinical Relevance**

Loss of cement at the margins of restorations can initiate a variety of clinical issues that may ultimately result in restoration loss and replacement.

#### SUMMARY

One of the primary areas of concern with luting agents is marginal gap erosion and attrition. The purpose of this laboratory study was to evaluate bulk and marginal slit (gap) generalized wear of self-adhesive resin cements. Three self-adhesive resin cements were used in this study: G-CEM LinkAce (LA), Maxcem Elite (ME), and RelyX Unicem2 Automix

Toshiki Takamizawa, DDS, PhD, assistant professor, Operative Dentistry, Nihon University School of Dentistry, Tokyo, Japan

\*Wayne W Barkmeier, DDS, MS, professor, General Dentistry, dean emeritus, Creighton University School of Dentistry, Omaha, NE, USA

Mark A Latta, DMD, MS, professor, General Dentistry, dean, Creighton University, Omaha, NE, USA

- Thomas P Berry, DDS, assistant professor, General Dentistry, School of Dentistry, Creighton University, Omaha, NE, USA
- Akimasa Tsujimoto, DDS, PhD, assistant professor Operative Dentistry, Nihon University School of Dentistry, Tokyo, Japan
- Masashi Miyazaki, DDS, PhD, professor and chair, Operative Dentistry, Nihon University School of Dentistry, Tokyo, Japan
- \*Corresponding author: General Dentistry, Creighton University School of Dentistry, 2500 California Plaza, Omaha, NE 68178, USA; e-mail: wbark@creighton.edu

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(RU). A custom stainless-steel fixture with a cavity 4.5 mm in diameter and 4 mm deep was used for simulated generalized (bulk) wear. For simulated marginal gap wear, a two-piece stainless-steel custom fixture was designed with a slit (gap) 300 µm wide and 3 mm in length. For both wear models, 20 specimens each for each of the three adhesive cements were made for both light-cure and chemicalcure techniques. The cured cements were polished with a series of carbide papers to a 4000-grit surface and subjected to 100,000 cycles using the slit (gap) wear model and 400,000 cycles for generalized (bulk) wear in a Leinfelder-Suzuki (Alabama machine) wear simulator (maximum load of 78.5 N). Flat-ended stainless-steel antagonists were used in a water slurry of poly(methylmethacrylate) beads for simulation of generalized contact-free area wear with both wear models. Before and after the wear challenges, the specimens were profiled with a Proscan 2100 noncontact profilometer, and wear (volume loss [VL] and mean facet depth [FD]) was determined using AnSur 3D software. Two-way analysis of variance (ANOVA) and Tukey post hoc tests were used for data analysis for the two wear models. Scanning electron microscopy (SEM) was used to examine polished surfaces of the resin cements and the worn surfaces after the wear challenges. The two-way ANOVA of VL using the generalized (bulk) wear model showed a significant effect among the three resin cement materials for the factor of resin cement (p < 0.001) and the interaction of the cement and cure method (p < 0.001), but not for the cure method (p=0.465). The two-way ANOVA for FD

also found a significant difference for the factor of resin cement (p < 0.001) and the interaction of the resin cement and cure method (p < 0.001), but not for the cure method (p=0.277). The simulated generalized (bulk) wear for the light-cure groups was as follows: VL (mm<sup>3</sup>): RU 0.631 (0.094), LA 0.692 (0.112), and ME 1.046 (0.141) and FD (µm): RU 43.6 (6.5), LA 47.0 (7.7), and ME 72.5 (9.9). The simulated generalized (bulk) wear for the chemical-cure groups was as follows: VL (mm<sup>3</sup>): LA 0.741 (0.105), RU 1.231 (0.234), and ME 1.305 (0.143) and FD (µm): LA 50.7 (7.2), RU 84.5 (16.1), and ME 91.7 (10.2). Simulated wear using the slit (gap) model for the light-cure groups was as follows: VL (mm<sup>3</sup>): RU 0.030 (0.006), LA 0.031 (0.006), and ME 0.041 (0.009) and FD (µm): RU 49.6 (5.7), LA 57.2 (8.4), and ME 70.9 (10.7). The wear values for the chemical-cure slit (gap) groups were as follows: VL (mm<sup>3</sup>): LA 0.031 (0.004), ME 0.038 (0.007), and RU 0.045 (0.009) and FD (µm): LA 53.9 (6.7), ME 63.5 (9.1), and RU 74.2 (12.9). Pearson correlation tests revealed a strong relationship between the two wear models for the light-cure groups and a good relationship for the chemical-cure groups. The observations using SEM showed differences in filler particle shape and size among the cements and the resultant effect of the wear challenges. The worn surfaces of each cement were essentially the same for both light-cure and chemical-cure methods. The bulk wear model and new slit (gap) model for evaluation of simulated generalized wear of luting agents demonstrated significant differences (p < 0.05)in relative wear among three self-adhesive resin cements and between visible light- and

# INTRODUCTION

chemical-cure techniques.

The evolution of adhesive dentistry procedures and materials has changed many facets of dentistry. The development of resin luting agents, along with adhesive dentistry techniques, has rapidly advanced the capability to bond indirect restorations to mineralized tooth structures and core buildup materials. One of the main advantages of resin cements, when compared to nonpolymer luting agents, is enhanced mechanical properties and the ability to adhesively bond to metal, ceramic, enamel, and dentin.<sup>1-8</sup> The use of etch-and-rinse bonding procedures along with resin luting agents has helped to promote the use of high-strength ceramic restorations. The more recent introduction of self-adhesive resin cements has reduced the required number of clinical steps in the bonding sequence, thereby reducing the number of treatment steps along with patient chair time. The dual curing capability (lightcure and chemical-cure) of many of the newer resin cements has extended their use to include restorations where light penetration to the intaglio surface is limited or not attainable. The use of resin cements is now also well established for fiber-reinforced composite materials and has extended into orthodontics for attachment of both metallic and ceramic brackets.

One of the challenges that remains, regardless of the cementing media, is marginal integrity.<sup>9</sup> A major concern with all dental cements is their ability to resist gap formation at the marginal closure area from attrition and erosion. Intact restoration margins reduce the potential for marginal staining, secondary caries, tooth sensitivity, and periodontal issues. Clinical criteria have been used to assess marginal integrity in long-term clinical trials. However, these studies are costly and take years to complete. Investigators have used laboratory marginal gap studies in an effort to assess the potential for cement loss at the margins of restorations.<sup>9-12</sup> These studies have shown an excellent relationship among 1) tooth margin and restoration gap width, 2) type of cement, and 3) cement wear. They found that enhanced wear resistance of resin cements is associated with a smaller filler particle size.

Depending on the clinical situation, dual-cure selfadhesive resin cements can be light-cured, chemically-cured, or can use a combination of light curing and chemical curing. The degree of polymerization conversion can also impact the wear resistance of a cement at the margin.<sup>13-15</sup> Less efficiency in polymerization is often seen in chemically-cured resin cements and may lead to higher wear.<sup>15</sup> Several selfadhesive resin cements are now available to the profession. The acidic monomers in these formulations can be a challenge when designing chemicalcure based polymerization components, as many amine initiators are quenched at low pH, leading to a lower degree of polymerization when compared to light curing.<sup>15-17</sup>

Table 1: Self-Adhesive R	esin Cements			
Material	Manufacturer	Lot	Shade	Study Code
G-CEM Link Ace	GC Corp (Tokyo, Japan)	1212144	A2	LA
Maxcem Elite	Kerr Corp (Orange, CA, USA)	4818000	Brown	ME
RelyX Unicem2 Automix	3M ESPE Dental Products (St Paul, MN, USA)	50153	A2	RU

Technology has rapidly advanced for assessing simulated laboratory wear by the introduction of noncontact optical profilometers and enhanced computer software for data analysis.<sup>18</sup> In addition, a new marginal slit (gap) model was developed for laboratory-generalized wear (contact-free area [CFA] wear) simulation in an effort to learn more about the wear resistance of newer-generation self-adhesive resin cements. The purpose of this study was to use the newly developed slit (gap) wear model and an established generalized (bulk) wear model to assess relative wear characteristics of newer dual-cure selfadhesive resin cements. The two hypotheses tested were the null hypotheses that 1) there will not be a significant difference ( $\alpha$ =0.05) in wear values among three dual-cured self-adhesive resin cements and that 2) wear of self-adhesive resin cements using light-cure or chemical-cure methods will not be different.

#### METHODS AND MATERIALS

Three self-adhesive resin cements (Table 1) were evaluated in this study: G-CEM LinkAce (LA) (GC Corp, Tokyo, Japan), Maxcem Elite (ME) (Kerr Corp, Orange, CA, USA), and RelyX Unicem2 Automix (RU) (3M ESPE Dental Products, St Paul, MN, USA). The evaluation components in this study included 1) simulated generalized (bulk) wear testing, 2) simulated marginal slit (gap) wear testing, 3) argon-ion etching scanning electron microscopy (SEM) of the resin cement surfaces, and 4) SEM examination of the cement wear facets.

### Generalized (Bulk) Wear Simulation

Forty specimens for each of the three self-etching resin cement materials (total of 120 specimens) were prepared for wear challenges of 400,000 cycles using a generalized (bulk) wear model (CFA wear) in a Leinfelder-Suzuki (Alabama) wear simulation device. The methodology for sample preparation and the generalized wear model was previously described by Barkmeier and others.<sup>18-20</sup> In summary, stainless-steel custom fixtures with cavities 4.5 mm in diameter and 4 mm deep were used to hold the resin cement materials. Twenty specimens for each resin

cement were light-cured in two increments of approximately 2 mm for 40 seconds with a Spectrum 800 curing unit (Dentsply Caulk, Milford, DE, USA) set at 600 mW/cm<sup>2</sup>. The other 20 specimens for each cement) were chemically-cured (the light curing unit was not used). After 24 hours, the cement surfaces were polished flat to 4,000 grit (Figure 1) using a sequence of silicon carbide papers (Struers Inc, Cleveland, OH, USA).

The custom fixtures were mounted inside a plastic water bath, and a brass cylinder was placed around each fixture. The water bath fixture was then attached to the wear simulator. A water slurry of poly(methylmethacrylate) (PMMA) was used as the abrasive media and placed inside the cylinders over the resin cement specimens.

Stainless-steel antagonists 6.5 mm in diameter (Figure 2), mounted in spring-loaded pistons, were then used to deliver the wear challenges in the wear simulation machine (Figure 3). The pistons rotated approximately 30 degrees as the load was applied (maximum load of 78.5 N) at a rate of 2 Hz and then



Figure 1. Custom stainless-steel fixture for generalized (bulk) wear.

Figure 2. Stainless-steel flat-ended antagonist.

Figure 3. Generalized wear model.

 Fig 2
 Fig 3

counterrotated back to their original position as the cycle was completed.

Prior to wear testing, the specimens for each resin composite material were profiled using a Proscan 2100 noncontact optical profilometer (Scantron Industrial Products, Ltd, Taunton, UK) with Proscan software. The individual scanned surfaces were used as the pretest digitized surface contour for each specimen.

Following the 400,000 cycling period, the specimens were ultrasonically cleaned (L&R Solid State Ultrasonic T-14B, South Orange, NJ, USA) for 3 minutes in distilled water and then again profiled using the Proscan 2100 unit. The X, Y, and Z coordinates of the before and after scans from the Proscan software were exported for analysis with AnSur 3D software (Minnesota Dental Research Center for Biomaterials and Biomechanics, University of Minnesota, Minneapolis, MN, USA).

Wear measurements were determined from differences between the before and after data sets. A computerized fit was accomplished on the before and after surface contours using AnSur 3D. Volume loss  $(VL)\,(mm^3)$  and facet depth  $(FD)\,(\mu m)$  of the wear areas were recorded for each generalized wear specimen.

### Slit (Gap) Model: Generalized Wear Simulation

A two-piece stainless-steel custom fixture was designed to examine resin cement wear using a thin slit or gap. This design was used in an attempt to simulate generalized wear (CFA wear) of resin cements at the marginal closure area. The overall fixture size was the same as the stainless-steel custom fixture used for generalized (bulk) wear, but a thin slit replaced the cylindrical cavity in the center of the top flat surface. The two-piece fixture was designed to have a slit (gap) 300  $\mu$ m wide, 3 mm in length, and 4 mm in depth (Figures 4 and 5). A 3% paraffin-in-hexane solution was used as a separating media on the interior walls of the two-piece fixture.

Twenty specimens each of the three self-adhesive resin cements were made for both light-cure and chemical-cure groups. The cured cements were polished and subjected to 100,000 cycles in the same manner as described above for generalized (bulk)

> Figure 4. *Slit (gap) fixture for generalized wear simulation.*

> Figure 5. Unassembled slit (gap) fixture.



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Fig 4

Table 2: Two-Way Analysis of Variance: SimulatedGeneralized (Bulk) Wear, Volume Loss					
Source	Sum of Squares	Degrees of Freedom	Mean Square	<i>F</i> -Ratio	p
Cement	10,449.9	2	5224.9	55.1	< 0.001
Cure method	51.1	1	51.1	0.538	0.465
Cement*cure method	33,925.2	2	16,962.6	178.7	<0.001

wear. The Proscan 2100 was used to make pretest and posttest scans, and the digitized surface contours were exported for examination with AnSur 3D software. Cement VL and FD were determined on the resin cement in the slit (gap) space as described above for the simulated generalized (bulk) wear model.

#### SEM

Specimens of each of the three self-adhesive resin cements were prepared for argon-ion etching and SEM examinations at Nihon University School of Dentistry (Tokyo, Japan). The three resin cements examined in this manner were not from the same lot numbers as the materials subjected to wear simulation studies and postwear SEM examinations at Creighton University School of Dentistry (Omaha, NE, USA). The lot numbers of the cement materials for the argon-ion etching SEM examinations were as follows: LA: 1402271, ME: 4394312, and RU: 497681.

The surfaces of the light-cure cements were polished to a high gloss with abrasive discs (Fuji Star Type DDC, Sankyo Rikagaku Co Ltd, Saitama, Japan) followed by a series of diamond pastes down to 0.25- $\mu$ m particle size (DP-Paste, Struers, Ballerup, Denmark). The polished surfaces were then subjected to argon-ion beam etching (IIS-200ER, Elionix, Tokyo, Japan) for 45 seconds with the ion beam directed at the polished surfaces (accelerating voltage of 1.0 kV, ion current density of 0.4 mA/cm<sup>2</sup>). The surfaces were then coated in a vacuum evaporator with a thin film of gold. Observations were made with a scanning electron microscope (FE-8000, Elionix) using an operating voltage of 10 kV and a magnification of 5000×.

SEM examinations were completed at Creighton University School of Dentistry on the wear facets of the three resin cement materials (light-cured and chemical-cured) following simulated generalized wear using the two wear models. After the wear analysis, representative samples of each material were sputter coated with gold and palladium using an Emitech SC7620 Mini Sputter Coater (Quorum

Table 3: Two-Way Analysis of Variance: SimulatedGeneralized (Bulk) Wear, Facet Depth					
Source Sum of Degrees Mean <i>F</i> -Ratio <i>p</i> Squares of Square Freedom					
Cement	1.97	2	0.986	49.8	< 0.001
Cure method	0.024	1	0.024	1.195	0.277
Cement*cure method	6.78	2	3.39	171.0	<0.001

Technologies, Ashford, UK). The coated wear specimens were then examined near the center of the wear facet with a TM3000 Tabletop Microscope (Hitachi-High Technologies Corp, Tokyo, Japan) using an accelerating voltage of 15 kV and magnifications of  $2,500 \times$  and  $5,000 \times$ .

### **Data Analysis of Simulated Wear**

A two-way analysis of variance (ANOVA) (factors: [1] cement material and [2] cure method) and Tukey *post hoc* tests were used for data analysis of VL and FD of both light-cure and chemical-cure groups for both wear models. In addition, Pearson correlation tests were used to determine the relationship between the two wear models for both VL and FD of the light-cure and chemical-cure groups.

## RESULTS

#### **Generalized (Bulk) Wear Simulation**

The two-way ANOVA of the generalized (bulk) wear data for both VL and FD showed a significant effect (p < 0.001) for the factor of resin cement and the interaction of the resin cement and cure method. There was not a significant effect for the factor of cure method for either VL (p=0.465) or FD (p=0.277). The two-way ANOVA values are shown in Tables 2 and 3.

The results of the simulated generalized (bulk) wear are presented in Table 4. The VL (mm<sup>3</sup>) and FD (µm) for the light-cure groups were as follows: VL: RU 0.631 (0.094), LA 0.692 (0.112), and ME 1.046 (0.141) and FD: RU 43.6 (6.5), LA 47.0 (7.7), and ME 72.5 (9.9). VL and FD for light-cure RU and LA were statistically similar (p>0.05). ME exhibited statistically (p<0.05) greater VL and mean FD than RU and LA for the light-cure cements.

The VL (mm<sup>3</sup>) and FD ( $\mu$ m) for the chemical-cure groups were as follows: VL: LA 0.741 (0.105), RU 1.231 (0.234), and ME 1.305 (0.143) and FD: LA 50.7 (7.2), RU 84.5 (16.1), and ME 91.7 (10.2). For the chemical-cure cements, RU and ME show statisti-

Table 4: Simulated Generalized (Bulk) Wear of Self-Adhesive Resin Cements $(n=20)^a$						
Resin Cement Light-Cure			Chemical-Cure			
	Volume Loss (mm <sup>3</sup> )	Facet Depth (µm)	Volume Loss (mm <sup>3</sup> )	Facet Depth (µm)		
RU	0.631 (0.094) aA	43.6 (6.5) aA	1.231 (0.234) aB	84.5 (16.1) aB		
LA	0.692 (0.112) aA	47.0 (7.7) aA	0.741 (0.105) bA	50.7 (7.2) bA		
ME	1.046 (0.141) bA	72.5 (9.9) bA	1.305 (0.143) aB	91.7 (10.2) aB		
<sup>a</sup> Lowercase letters in vertical columns are not different at the 5% significance level. Same uppercase letters between columns indicate no difference (5% significance level) in light-cure vs chemical-cure of the same cement.						

cally (p < 0.05) greater VL and FD than LA. Light curing of the RU and ME resin cements appeared to be more effective than chemical curing in limiting simulated generalized (bulk) wear. The FD and VL of RU were statistically greater (p < 0.05) and nearly double for chemical-cure RU resin cement when compared to light curing. When comparing LA generalized wear of light-cure and chemical-cure cement, the results were statistically (p > 0.05)similar for both VL and FD.

# Slit (Gap) Model: Generalized Wear Simulation

The two-way ANOVA for both VL and FD using the slit (gap) model for generalized wear showed a significant effect for the factors of resin cement (p<0.001), cure method (p=0.002), and the interaction of resin cement and cure method (p<0.001). The two-way ANOVA values are shown in Tables 5 and 6.

The results of the slit (gap) model generalized wear are presented in Table 7. The VL (mm<sup>3</sup>) and FD (µm) for the light-cure groups were as follows: VL: RU 0.030 (0.006), LA 0.031 (0.006), and ME 0.041 (0.009) and FD: RU 49.6 (5.7), LA 57.2 (8.4), and ME 70.9 (10.7). VL in the slit model for light-cure RU and LA was statistically similar (p>0.05). ME exhibited statistically (p<0.05) greater VL than RU and LA for the light-cure cements. RU exhibited the least slit (gap) model FD wear of the light-cure cements.

The slit model VL  $(mm^3)$  and FD  $(\mu m)$  for the chemical-cure groups were as follows: VL: LA 0.031

Table 5:         Two-Way Analysis of Variance: Slit (Gap) Model         Generalized Wear, Volume Loss         V					
Source	Sum of Squares	Degrees of Freedom	Mean Square	<i>F</i> -Ratio	p
Cement	0.0015	2	0.000749	14.916	< 0.001
Cure method	0.000529	1	0.000529	10.541	0.002
Cement*cure method	0.0016	2	0.000828	16.495	<0.001

(0.004), ME 0.038 (0.007), and RU 0.045 (0.009) and FD: LA 53.9 (6.7), ME 63.5 (9.1), and RU 74.2 (12.9). The VL and mean FD of chemical-cure LA were significantly less (p<0.05) than RU and ME. RU exhibited the greatest VL and FD (p<0.05) when the chemical-cure groups were compared to light-cure groups while those of both LA and ME were essentially the same (p>0.05).

The results of the Pearson correlation tests are presented in Table 8. A strong relationship was found between the generalized (bulk) wear model and the slit (gap) model for the light-cure groups for VL (r=0.999) and FD (r=0.968). A good relationship was found between the two wear models for the chemical-cure groups for VL (r=0.799) and FD (r=0.752).

# **SEM Observations**

The ultrastructure examinations with argon-ion etching SEM revealed morphological differences in filler components of the cements (Figure 6a–c). All three resin cements exhibited a wide variety of filler particle sizes and shapes. The particle size distribution of ME appeared to include larger particles than either LA or RU. The ultrastructure micrographs demonstrated that the filler components were different, and these compositional differences may have influenced the wear characteristics of these materials.

The SEM exanimations of the worn surfaces of all three of the resin cements showed evidence of particle loss (plucking) from the simulated gener-

Table 6: 7	Two-Way Analysis of Variance: Slit (Gap) Model Generalized Wear, Mean Facet Depth					
Source	Sum of Squares	Degrees of Freedom	Mean Square	<i>F</i> -Ratio	p	
Cement	3143.047	2	1571.524	19.745	< 0.001	
Cure method	813.49	1	813.490	10.221	0.002	
Cement*cure method	5637.538	2	2818.769	35.416	<0.001	

Resin Cement Light-Cure		Cure	Chemical-Cure	
	Volume Loss (mm <sup>3</sup> )	Facet Depth (µm)	Volume Loss (mm <sup>3</sup> )	Facet Depth (µm)
RU	0.030 (0.006) aA	49.6 (5.7) aA	0.045 (0.009) bB	74.2 (12.9) aB
LA	0.031 (0.006) aA	57.2 (8.4) bA	0.031 (0.004) aA	53.9 (6.7) bA
ME	0.041 (0.009) bA	70.9 (10.7) cA	0.038 (0.007) cA	63.5 (9.1) cA

alized wear with both wear models (Figures 7a-d, 8a-d, and 9a-d). There were also microcracks on the resin surface that most likely resulted from wear challenge fatigue stress. There was no apparent difference in worn surface morphology between the two wear models or when comparing light-cure and chemical-cure surfaces for each cement (Figures 7a-d, 8a-d, and 9a-d). Any observed differences between the light-cure and chemical-cure surfaces, as well as comparisons between the bulk and slit model worn surfaces, were subtle. There did appear to be a qualitative morphological difference for RU, where the chemical-cure surfaces seemed to have more filler particle plucking than light-cure surfaces. This observation is consistent with the differences in wear values for RU between light-cure and chemical-cure specimens (Tables 9 and 10).

#### DISCUSSION

Two methods of wear simulation were used in this laboratory study to assess the relative wear resistance of three self-adhesive resin cements. Wear generated using a new slit (gap) model was compared to a commonly used simulated generalized (bulk) wear method. In both models, a flatended stainless-steel antagonist was used to produce wear using a water slurry of PMMA beads as the abrasive media. Both light-cure and chemicalcure specimens of the three self-adhesive resin cements were assessed using SEM examinations and wear analysis. The rank order (RU-LA-ME) of wear (VL and FD) for the three resin cements was the same for the two test models when the cements were light-cured (Tables 4 and 7). The rank order of

Table 8:	8: Pearson's Correlations (r): Generalized (Bulk) and Slit (Gap) Wear Models			
Polymerization Method		Volume Loss	Facet Depth	
Light-cure		0.999	0.968	
Chemical-o	cure	0.799	0.752	

wear using the chemical-cure method was not the same as the light-cure groups when the results of the two wear methods were examined (Tables 4 and 7).

The wear data of the resin cements for both the simulated (bulk) wear model and the slit (gap) model showed that the LA cement, when light-cured and chemical-cured, were similar and not statistically different (p>0.05) for FD and VL (Tables 4, 7, 9, and 10). The generalized (bulk) wear values of RU nearly doubled with the chemical-cure method when compared to light curing alone (Tables 4 and 9). The results of the slit (gap) model wear (VL and FD) of RU showed a 50% increase for the chemical-cure group when compared to light curing (Table 10). ME exhibited the highest wear values of the three materials in this study with both wear models (Tables 4 and 7), showing about a 25% increase in both VL and FD with the generalized (bulk) wear method (Table 9). A decrease in wear (VL and FD) for ME was found with the slit (gap) model for chemical curing when compared to light curing (Table 10).

Previous studies utilizing the generalized (bulk) wear model have used 400,000 cycles for testing of resin-based materials.<sup>18-23</sup> Leinfelder and Suzuki<sup>22</sup> have reported that for resin composite materials, there was a high level of agreement between wear generated with 400,000 cycles in the Alabama simulator and 3 years of clinical service. Over the years, most of the laboratory testing with the Alabama machine has been done using 400,000 cycles.

With the new slit (gap) model utilized in this study, 100,000 cycles were used for testing versus the 400,000 cycles. There was a twofold reason for reducing the number of cycles with the slit (gap) test model in the preliminary or initial testing of the new wear model: 1) force concentration from the antagonist was applied to a much smaller area of resin cement than the traditional generalized (bulk) model, and 2) testing was expedited when compared to using 400,000 cycles. The



Figure 6a. G-CEM Link Ace: argon-ion etched surface (5000×).

Figure 6b. Maxcem Elite: argon-ion etched surface (5000×).

Figure 6c. Rely X Unicem 2 Automix: argon-ion etched surface (5000×).

results clearly showed that the slit (gap) model could discriminate wear among the three selfadhesive resin cements using 100,000 cycles (Table 7). Previous studies<sup>24,25</sup> comparing degree of conversion for light curing and chemical curing have generally found that light curing produces a significantly higher degree of conversion than chemical curing alone for dual-cure cements. Ferracane and others<sup>13,26</sup> have also reported that wear resistance and mechanical properties of resin composites are increased by improving the degree of conversion. The ability of dual curing cements to effectively cure in the chemical set mode for indirect restorations is vitally important to the long-term success of restorative procedures where light curing is not possible or is limited.

A critical factor in the setting reaction for chemical curing of self-adhesive resin cements is the influence of the amine initiator. It is speculated from earlier studies showing a superior degree of conversion of dual-cured cements with photoactivation compared to chemical curing that this difference was caused by acidic monomers impacting negatively on the chemical-cure setting reaction by lowering the pH.<sup>15,24,25</sup> In examining simulated generalized (bulk) wear in the present study, two of the three self-etch adhesive systems (RU and ME) showed significantly (p < 0.05) less wear (VL and FD) for light-cure groups when compared to chemical-cure groups (Table 4). The simulated (bulk) wear of LA was slightly greater in the chemical-cure group when compared to the light-cure material (Table 4), but this difference was not significant (p>0.05). The wear of LA in the slit (gap) model was essentially the same for the light-cure and chemical-cure groups. The wear of RU in the slit (gap) model (Table 7) exhibited the same pattern as the generalized (bulk) model (Table 4) with the chemical-cure cement showing significantly (p < 0.05) more wear than the lightcure cement. The wear of ME was slightly less (p>0.05) in the chemical-cure group in the slit (gap) model (Table 7) when compared to the lightcure counterpart. It is interesting to note that the wear of chemically-cured LA in both the generalized (bulk) wear model and the slit (gap) wear model was significantly less than both RU and ME (Tables 4 and 7). This would suggest that the

Table 9:       Generalized (Bulk)       Wear Model: Percent Change, Light-Cure to Chemical-Cure (n=20)						
Resin Cement		Volume Loss (mm <sup>3</sup> )			Facet Depth (µm)	
	Light-Cure	Chemical-Cure	% Change	Light-Cure	Chemical-Cure	% Change
RU	0.631 (0.094)	1.231 (0.234)	95.1	43.6 (6.5)	84.5 (16.1)	93.8
LA	0.692 (0.112)	0.741 (0.105)	7.1	47.0 (7.7)	50.7 (7.2)	7.9
ME	1.046 (0.141)	1.305 (0.143)	24.8	72.5 (9.9)	91.7 (10.2)	26.5



Figure 7a. *G-CEM Link Ace/light-cure: generalized (bulk) wear (400,000 cycles) near center of wear facet (2500×).* 

Figure 7b. G-CEM Link Ace/chemical-cure: generalized (bulk) wear (400,000 cycles) near center of wear facet (2500×).

Figure 7c. *G-CEM Link Ace/light-cure: slit (gap) generalized wear (100,000 cycles) near center of wear facet (2500×).* 

Figure 7d. G-CEM Link Ace/chemical-cure: slit (gap) generalized wear (100,000 cycles) near center of wear facet (2500×).



x2.5k 30 um

polymerization reaction with chemical curing for LA is more effective than RU and ME. Thus, the null hypotheses—1) there will not be a significant difference in wear values among three dual-cured self-adhesive resin cements and 2) wear of selfadhesive resin cements using light-cure or chemical-cure methods will not be different—are rejected for RU and ME in the generalized (bulk) wear model but not for LA and rejected for RU in the slit (gap) wear model.

Belli and others<sup>14</sup> reported data for self-adhesive resin cements using a gap model for both toothbrush abrasion and the ACTA wear method. They related that self-adhesive resin cements exhibited good wear resistance to toothbrush abrasion but showed much more wear under the heavier loading with the ACTA test. They also reported that no correlation ( $R^2$ =0.0567) was found between the two test methods. In the present study comparing x2.5k 30 u

the generalized (bulk) wear model and the slit (gap) model, a strong correlation (Pearson) was found between the light-cure groups for both VL (r=0.999) and FD (r=0.968). For the chemical-cure groups, the correlations between the two models were r=0.799 for VL and r=0.752 for FD. While the associations for the chemical-cure groups were not as robust as for the light-cure groups, the relationships were good for the chemical-cure groups with the two wear methods used in this study.

Barkmeier and others<sup>18-20</sup> have conducted several studies with the generalized (bulk) wear model used in this study to evaluate the wear resistance of resin composite materials. The new slit or gap model was used in the present study to more closely parallel a clinical situation in the assessment of wear resistance of self-adhesive resin cements. The new slit (gap) model delivers the wear challenges with the

Table 10:       Slit (Gap) Wear Model: Percent Change, Light-Cure to Chemical-Cure (n=20)						
Resin Cement	in Cement Volume Loss (mm <sup>3</sup> )				Facet Depth (µm)	
	Light-Cure	Chemical-Cure	% Change	Light-Cure	Chemical-Cure	% Change
RU	0.030 (0.006)	0.045 (0.009)	50.0	49.6 (5.7)	74.2 (12.9)	49.6
LA	0.031 (0.006)	0.031 (0.004)	0.0	57.2 (8.4)	53.9 (6.7)	-5.8
ME	0.041 (0.009)	0.038 (0.007)	-7.3	70.9 (10.7)	63.5 (9.1)	-10.4



Figure 8a. Maxcem Elite/light-cure: generalized (bulk) wear (400,000 cycles) near center of wear facet (2500×).

Figure 8b. Maxcem Elite/chemical-cure: generalized (bulk) wear (400,000 cycles) near center of wear facet (2500×).

same stainless-steel antagonist tip used in the generalized (bulk) wear simulation model. The primary difference is the area of cement exposed to the wear process. Overall, the correlation of VL and FD using the two methods was excellent. While the goal of using the slit (gap) model was to more closely replicate the type of abrasion that may occur in the oral cavity, the results indicate that the wear resistance of resin cements can be assessed with the standard generalized (bulk) wear model. The slit (gap) method does not appear to offer any real advantages in the assessment of the wear resistance

advantages in the assessment of the wear resistance of self-adhesive resin cements. However, the slit (gap) model reaffirms that abrasive wear of a thin film of a resin cement in marginal closure areas remains an issue for long-term clinical performance of cemented restorations.

Wear resistance of resin cements may be influenced by water sorption of these materials. Ferracane and others<sup>1</sup> have cautioned that the hydrophilic nature of resin cements, due to the low pH of cured material, can result in excessive water sorption, which may cause material swelling and compromised mechanical properties. These authors indicated that the concentration of acidic monomers must be balanced to effectively etch mineralized tooth structures for bonding but also avoid hydrophilicity in the cured cement. Studies have shown that some resin cements are more prone to water sorption and subsequent degradation than others.<sup>16,27-31</sup> Zorzin and others<sup>16</sup> have related that resin cement specimens that are desiccated during the acid-base reaction phase could result in extraction of water produced during the setting reaction and that this could interfere with setting and pH neutralization kinetics. In the present laboratory study, all the specimens were fabricated on the bench and stored at room temperature for 24 hours before polishing and testing. The materials were rehydrated during the wear-testing procedure. However, in future studies, the storage conditions and resultant effects on these types of specimens, especially the chemical-cure groups, should be investigated. The wear mechanics, as related to hydration during the setting reaction and subsequent water sorption, needs further attention. Another potential influence in wear characteristics using the slit (gap) model

Figure 8c. Maxcem Elite/light-cure: slit (gap) generalized wear (100,000 cycles) near center of wear facet (2500×).

Figure 8d. Maxcem Elite/chemical-cure: slit (gap) generalized wear (100,000 cycles) near center of wear facet (2500×).



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would be the effect of promoting adhesion to the internal surfaces of the chamber in the slit (gap) specimen holder. Eliminating the separating medium and including a procedure to promote bonding between the cements and the stainless-steel fixtures may improve adhesion and more effectively mimic clinical situations. This procedural change may impact of the simulated wear values in the slit (gap) model and should be investigated further in future studies.

#### CONCLUSIONS

A generalized (bulk) wear simulation model and a new slit (gap) wear model showed differences (p < 0.05) in the relative wear resistance of three self-adhesive resin cements and between light cure and chemical-cure techniques. Both wear models provided valuable information regarding the wear resistance of self-adhesive resin cements.

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#### **Regulatory Statement**

This study was conducted at the Creighton University School of Dentistry and Nihon University School of Dentistry.

**Conflict of Interest** 

The authors of this article 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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Figure 9a. RelyX Unicem2 Automix/ light-cure: generalized (bulk) wear (400,000 cycles) near center of wear facet (2500×).

Figure 9b. RelyX Unicem2 Automix/ chemical-cure: generalized (bulk) wear (400,000 cycles) near center of wear facet (2500×).

Figure 9c. RelyX Unicem2 Automix/ light-cure: slit (gap) generalized wear (100.000 cycles) near center of wear facet (2500×).

Figure 9d. RelvX Unicem2 Automix/ chemical-cure: slit (gap) generalized wear (100,000 cycles) near center of wear facet (2500 $\times$ ).

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