

SMART *CEM*TM2
SELF-ADHESIVE CEMENT

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1. Product Description & Features

SmartCem™2 Self-Adhesive Cement is a two-component, dual-cure, high strength self-adhesive cement containing fluoride fillers. SmartCem™2 combines esthetic shading with a self-etching adhesive, making it suitable for the permanent cementation of metal, PFM, resin/composite, ceramic and porcelain inlays, onlays, crowns and bridges and endodontic posts without application of a separate dentin/enamel adhesive bonding agent/system. Cured SmartCem™2 is essentially hydrophobic, minimizing post-cure water sorption, solubility and hygroscopic expansion. SmartCem™2 is available in a convenient dual-barreled syringe or in the digit® Targeted Delivery System unit dose cartridge to simplify delivery and minimize product waste.

While the features of SmartCem™2 are described in this manual, a partial list is included below.

- Low Film thickness
- Low Solubility & Expansion
- A *new* Initiating system providing:
 - Improved Stability with consistent work/set times
 - No refrigeration required
- 5 Color-stable Shades in ALL deliveries
- Fluoride Containing glass filler

2. Composition

As shown in Figure 1, the composition of SmartCem™2 comprises both familiar and new components.

Figure 1.
The composition of SmartCem™2 comprises familiar and new components, each having specific functions in the overall composition.

Composition	
Components	Function
➤ UDMA Resin	➤ Structural Resin
➤ EBPADMA Urethane Resin	➤ Structural Resin
➤ Di- and tri-functional diluents	➤ Crosslinking agents-- Strength
➤ PENTA	➤ Adhesion promoter
➤ Proprietary photoinitiating system	➤ New, acid resistant system; Long shelf-life
➤ Proprietary self-cure initiating system	➤ New, acid resistant system; Stable shelf-life
➤ 69% fillers by wt., 46% by vol.	➤ Fluoride Glass

The polymerizable resins are used in other Dentsply products, such as Esthet•X® Micro-matrix Restorative, TPH® 3 Micro Matrix Restorative, Xeno® IV Dual Cure Self-Etching Dental Adhesive, etc. These resins provide structural reinforcement of the resin cement, as well as providing strong crosslinking of the polymer network upon polymerization. As a result, the mechanical and physical properties of the SmartCem™2 are enhanced and strengthened. The adhesion promoter, PENTA, is well known to Dentsply adhesive products, including Prime & Bond® NT™ Universal Dental Adhesive System, Xeno® IV, etc. This phosphoric acid modified monomer has been demonstrated to interact directly with the calcium in tooth structure, resulting in strong covalent bonding to the tooth surface¹. The new, proprietary ingredients include the photoinitiator system, as well as the self-cure reactive components of the dual cure material (see section 5.4). The glass filler (69% by weight, 46% by volume) used in the product contains fluoride fillers, similar to those used in other restorative products noted above.

3. Indications for Use

SmartCem™2 is intended for the cementation of indirect restorations including ceramic, composite and metal-based inlays, onlays, crowns, bridges, and posts. SmartCem™2 is contraindicated for use with patients who have a history of severe allergic reaction to methacrylate resins or any of the components. SmartCem™2 is contraindicated for direct application to dental pulp tissue (direct pulp capping). (See Appendix A for complete Directions for Use.)

4. Shades

SmartCem™2 is available in 5 shades: translucent, light, medium, dark, and opaque.

5. In Vitro, Clinically Relevant Physical Properties

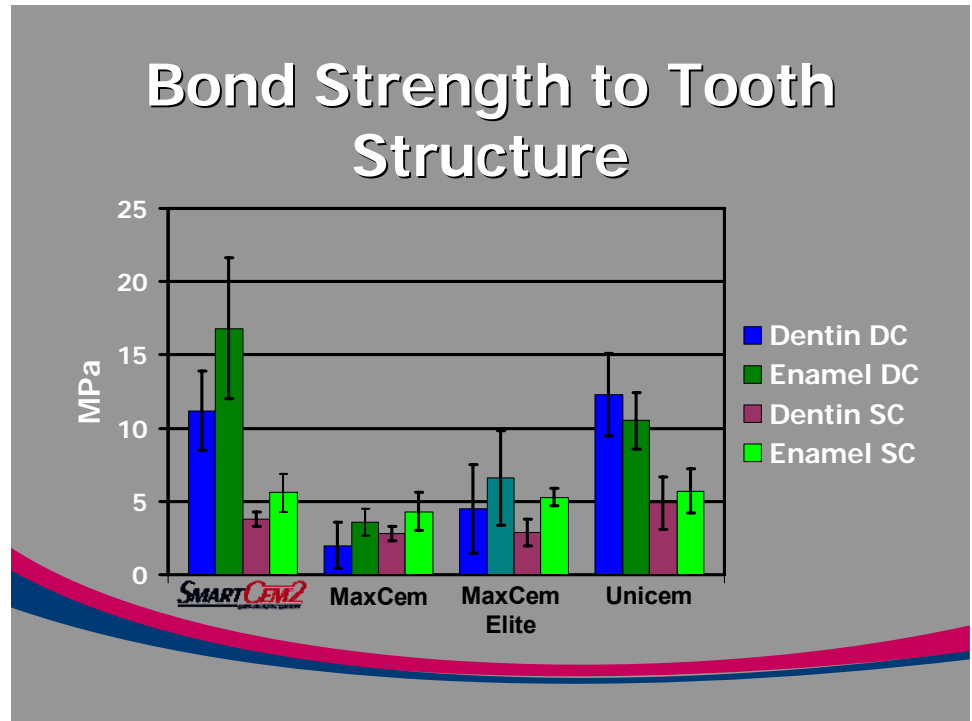
The data presented in the following sections represents those *in vitro* test procedures that are designed to closely approximate clinically relevant properties of the SmartCem™2 cement. All results presented were performed in the same laboratories under identical conditions wherever possible. Thus, within each group of test results, comparison among products may be inferred. Caution should be applied when attempting to compare similar test results from different laboratories due to potentially different test conditions, parameters, etc. Where noted, accepted, standardized ISO test methods were utilized in performing the testing. Please refer to the appropriate Appendices for a description of the complete test methods and methodologies used for each respective property noted.

5.1. Adhesion To Tooth Structure

5.1.1. Shear Bond Strength to Dentin & Enamel

The bond strengths to dentin and enamel were measured for several self-adhesive cements and are recorded in Figure 2.

Figure 2.
Shear bond strengths to human enamel and dentin using self-adhesive cements, both in the dual cure (DC) and self-cure (SC) mode.

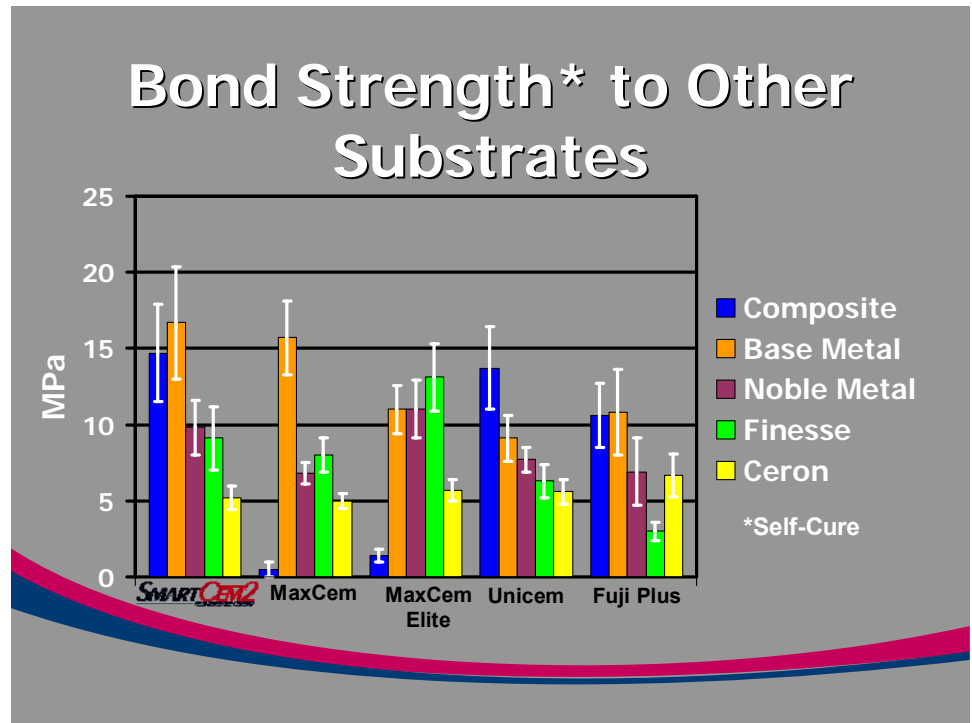


As shown in the figure, both the dual cure (including exposure with visible light) and the self-cure (no exposure to visible light) modes were examined. As has been noted with previous dual cure materials, exposure to visible light as part of the curing process generally leads to higher bond strengths, due to the more energetic conditions of curing. In addition, conditions used in the laboratory to prepare and measure self-curing specimens often lead to more variability in the test procedure, which can result in diminished bond strength results. As noted in the Figure 2, there are two distinct groups: SmartCem™2 and Rely X™ Unicem are roughly equivalent, while MaxCem™ and MaxCem™ Elite represent a separate group.

5.1.2. Shear Bond Strength to Various Substrates

The shear bond strength to various substrates was measured for several self-adhesive cements, as displayed in Figure 3.

Figure 3.
The shear bond strengths of SmartCem2 and other cements (self-cure only) to various substrates.



It is important to understand the adhesive quality of a self-adhesive cement to differing materials that may be used as substrates to which the cement is bonded. Thus, a representative sampling of 5 different materials was tested for adhesion using the cements listed in Figure 3. As shown in the figure, the results for the cements tested produced similar values for the various substrates, with the notable exception of the very low bond strength of MaxCem™ and MaxCem™ Elite to self-cure composite.

5.2. Crown Retention

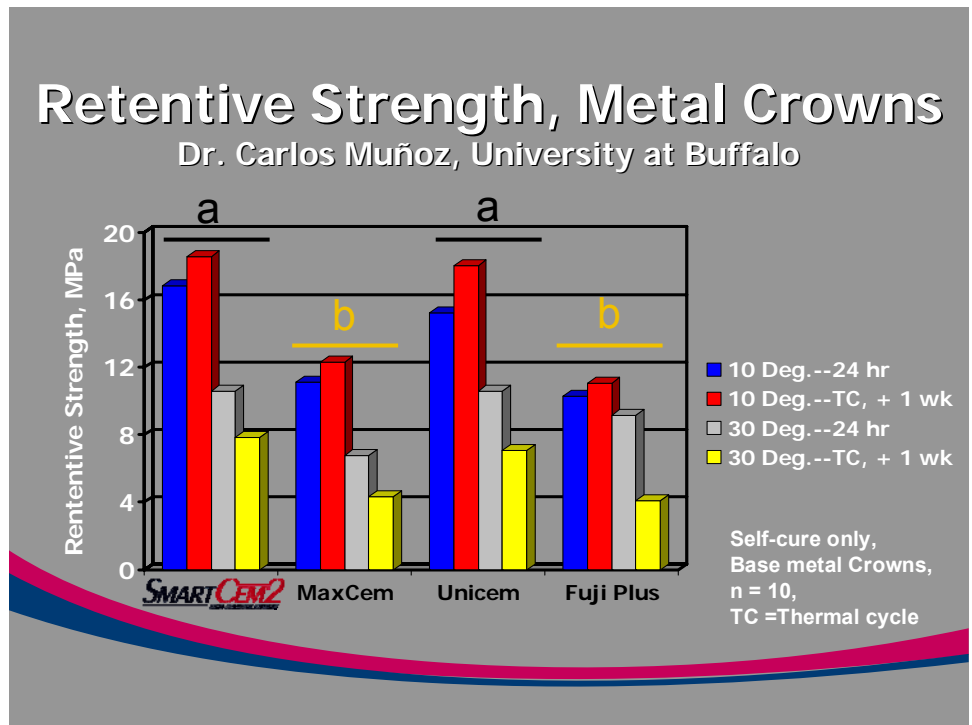
As illustrated above, the bond strength of SmartCem™2 to enamel and dentin, as well as various substrates, was determined. To further define the effectiveness of the cement under clinically relevant conditions, the retention of crowns cemented to extracted human teeth was

measured. Three different test sites were utilized in performing this test, the results of which are shown below. It should be noted that comparing actual numerical results from laboratory to laboratory is not possible due to different testing protocols and parameters. However, the relative results within each laboratory may be considered.

5.2.1. Metal Crowns, Dr. Carlos Muñoz

The purpose of this study was to evaluate the retention of crowns cemented with various self-adhesive cements and a glass ionomer control. The results are presented in Figure 4.

Figure 4.
Retentive strengths (crown-pull test) of metal crowns cemented to extracted human molars of varying degree of taper.



Natural tooth preparations with tapers of 10 and 30 degree of Total Occlusal Convergence (TOC) 4 mm in height were prepared. Eighty restorations with each taper were made. Metal crowns made of a base metal alloy were fabricated and luted with the cements. The cemented crowns were tested using a tensile force at: 1) 24 hours after cementation and 2) 1 week stored at 37 deg C @ 100% relative humidity and 500 thermal cycles.

The retentive bond strength was measured using an MTS Universal Testing machine (1125) at a crosshead speed of 0.1 cm/min. The load required to debond the specimens was

recorded in Kg and the mean retentive strength of the ten specimens calculated and reported in mega-pascals using the following equation:

$$\text{Tensile Bond Strength (MPa)} = \text{force (Kg)} / \text{Surface area (cm}^2\text{)} \times 0.09807.$$

The last number was used to convert kg/cm² to MPa. The surface area of each specimen was calculated as the sum of the conical surface of the frustum and the surface area of the top of the frustum. The retention strength for each preparation for the luting cements, aging and TOC were analyzed by using a three-way analysis of variance (ANOVA). All tests were conducted at alpha = 0.5. Following testing of the specimens, the mode of failure was classified into one of three groups by visual and light microscope inspection. The three groups were:

- A.- Cohesive failure, where the failure occurred either completely within the tooth or completely within the luting agent
- B.- Adhesive failure, where the bond fails between the composite resin and the tooth with no cement left on the tooth and or the crown.
- C.- Fracture of the crown and/or tooth.

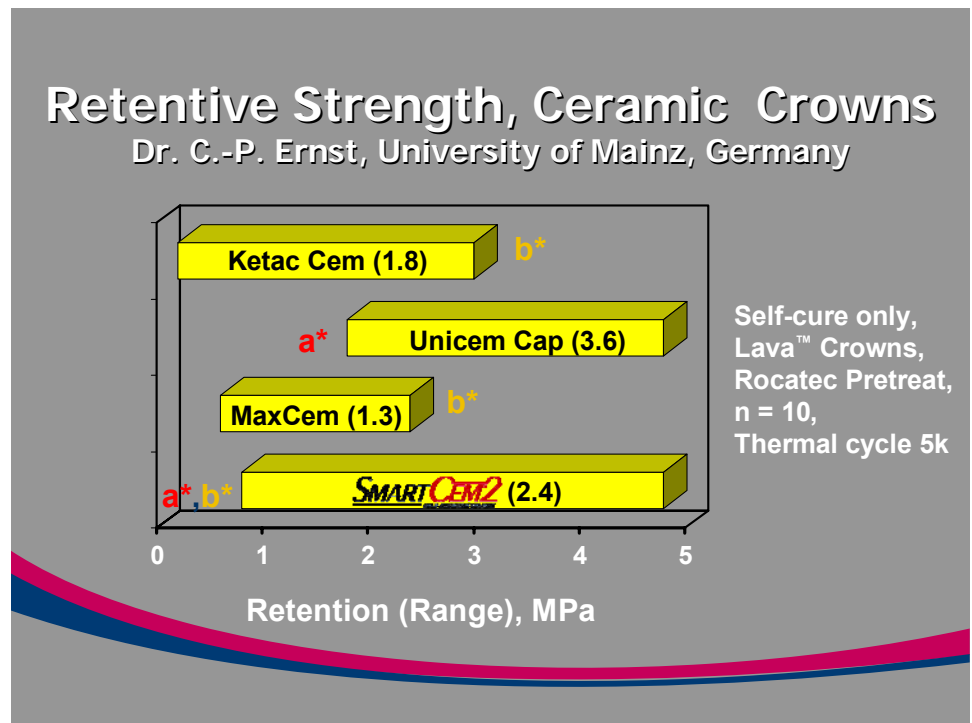
A three way ANOVA indicates that there was significant difference among two of the main effects: cements and taper ($p < 0.001$) in regards to the retentive strength and no significant difference for time ($p > 0.660$). The mean retentive strength of the SmartCem™2 and Unicem was statistically higher than that of Maxcem and Fuji regardless of the time that the cements were tested $p < 0.001$. When compared, there was no statistical difference among the SmartCem™2 and Unicem ($p = 0.518$) and between Maxcem and Fuji ($p = 0.779$). There was a statistical difference among cements when the tapers were compared. All cements showed a decrease in retentive strength when the crowns were cemented using a 30 degree taper preparation ($p = 0.030$).

The results of the study indicated that there was a difference between the retention of the different cements. Maxcem, which is a self-adhesive resin cement, and Fuji Plus, which is a glass ionomer, measured similar retention values regardless of the TOC or storage time. The values for these two cements were about 40% lower than those obtained for the SmartCem™2 and for Unicem, both self-adhesive resin cements.

5.2.2. Ceramic Crowns, Dr. C-P. Ernst

The aim of the study was to evaluate the retentive strength of zirconium-oxide crowns cemented with several self-adhering cements, including Rely X™ Unicem, MaxCem™, and SmartCem™ 2. See Figure 5.

Figure 5.
Retentive strength (crown-pull test) of ceramic crowns cemented to extracted human teeth. Force to remove measured after 5000 thermal cycles.



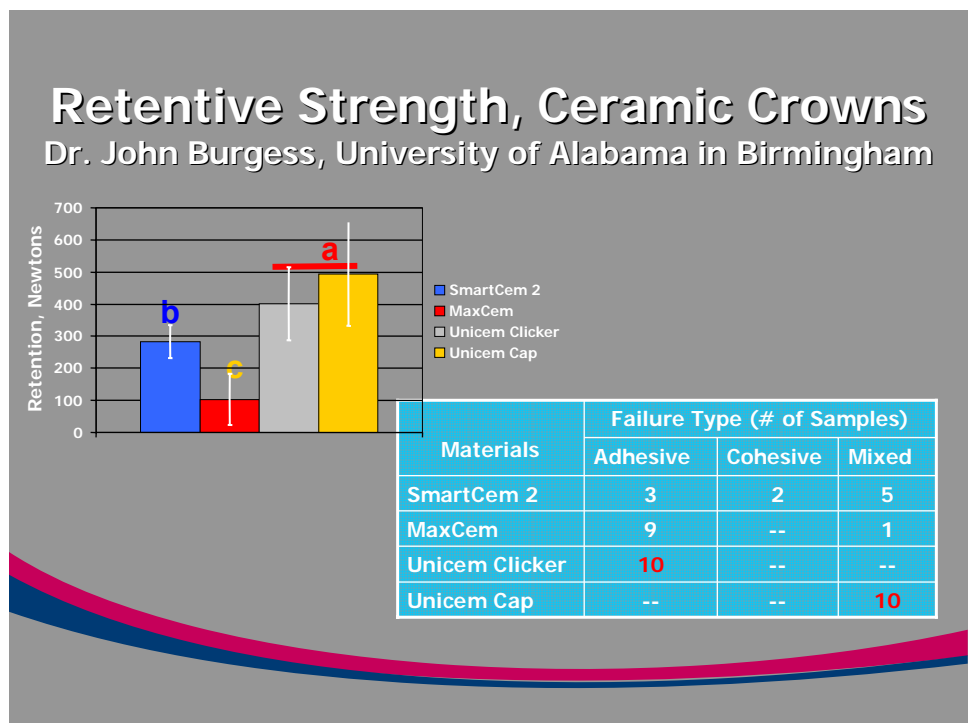
The adhesive cement systems were used according to manufacturers' recommendations; in dual-curing systems, only the self-curing mode was conducted. The crowns' inner surfaces were sandblasted (Rocatec Pre). After thermal cycling (5000 x, 5/55°C), the cemented zirconium-oxide LAVA crowns (Rocatec-pre-treatment at the outer surface; connected over a low shrinkage epoxy resin to macro-mechanical undercuts in a resin block made out of

Paladur denture base material) were removed along the path of insertion (Zwick 1425). The retention surface was determined individually for each tooth. Statistical analysis: Wilcoxon test/Bonferroni adjustment, 5% level. Results: The retentive strength values [N/mm²] were (Min/Q1/Median/Q3/Max): Rely X™ Unicem: 1.8/2.6/3.6/4.3/4.7; MaxCem™: 0.6/0.9/1.3/1.6/2.3; SmartCem™2: 0.8/1.3/2.4/3.1/4.8; Ketac Cem: 0.2/1.0/1.8/2.2/3.0. A wide variability of median retentive strength values was found within the group of self-adhering cements.

5.2.3. Ceramic Crowns, Dr. John Burgess

This study also involved zirconia crowns using self-adhesive resin cements SmartCem™2, MaxCem™, Rely X™ Unicem Aplicaps, and Rely X™ Unicem Clicker. The results are presented in Figure 6.

Figure 6.
Retentive strength (crown-pull test) of ceramic crowns cemented to extracted human teeth. Force to remove and failure mode were measured after 24 hours.



Extracted teeth with notched roots were retained in cylinders filled with acrylic resin. The occlusal surfaces were ground flat and placed into a lathe for precise uniform reduction with diamond cutting tools to produce a uniform crown preparation with exact taper, diameter and

fit. An orientation groove was placed into the occlusal surface of preparation by hand using a 69 L bur and high-speed hand piece. After the teeth were prepared to uniform dimensions, zirconia crowns were waxed and milled. After sintering, the finished crowns were individually fit on the tooth, margins checked for opening and fit (explorer does not catch) and the crowns cemented. The cements were mixed following the manufacturers directions and a 2kg weight was placed on the cemented crown until the cement had set. Excess cement was carefully removed and the crowns were allowed to set in tap water for 24 hours before debonding. A metal rod was placed through the hole on the crown and through the loops of a wire. Specimens were attached to the hook of the testing machine using the wire (INSTRON Model no: 5565) and loaded in tension at a cross-head speed of 0.5 mm/min until debonding occurred. The force (N) of debonding was recorded. Examination of the failure site was made visually and recorded as cohesive, mixed or adhesive. The results shown in Figure 6 were analyzed with ANOVA and Tukey B post hoc test.

Three significantly different groups were present. MaxCem™ produced significantly lower tensile strengths than any other cement. The SmartCem™2 was intermediate and the Unicem produced the highest tensile strengths. Rely X™ Unicem Clicker and capsule were not significantly different from one another. However, examination of the results for the Rely X™ Unicem and Clicker showed completely different modes of failure, with the Clicker showing exclusively adhesive failures, while the failures with the Rely X™ Unicem Aplicaps were completely mixed.

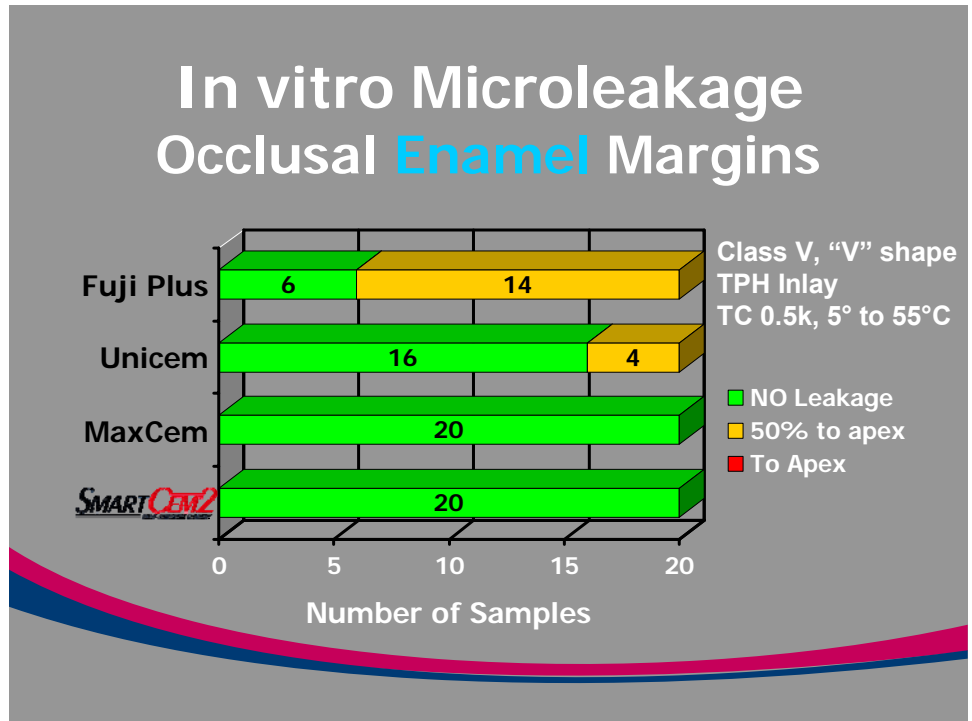
5.3. Microleakage

The extent to which a cement is able to seal the marginal areas around an indirect restoration can be demonstrated through the use of the in vitro test for resistance to microleakage. In this test, non-carious human molars were used. Two class V cavity preparations (“v”-shaped) were made on opposite sides of each non-carious human molar with the occlusal margins in enamel and gingival margins in cementum. Approximate dimensions of the preparations were 4 mm mesio-distally, and 3 mm occluso-gingivally, and 2 mm pulpal.

TPH®3 A3.5 was used to fabricate non-cemented, custom inlays for each restoration. The TPH®3 material was cured using a Spectrum 800 curing light for 40 second, and the resulting inlay was removed from the cavity preparation. The appropriate cement was applied to the TPH®3 inlay and custom inlay was re-inserted into each cavity. The restoration was light cured a total of 40 seconds with a Spectrum® 800 at 550 mw/cm² or allowed to self-cure for 15 minutes in the absence of light. The restoration margins were finished and polished with a Pogo® Diamond Micro-Polisher. Thereafter, the samples were stored in 37°C deionized water for 24 hours, followed by thermal cycling (540 cycles between 55°C and 5°C). Following the thermal cycling period, the teeth were evaluated for marginal microleakage using a silver nitrate staining technique. Leakage along the gingival wall of the cavity was scored according to the following criteria: a) 0- no dye penetration; b) 0.5- dye penetration to one-half of the distance to the apex.; c) 1- leakage to the bottom (apex) of the cavity; d) 1.5- penetration past the apex and observed on the occlusal wall to the 1/2 the length of the occlusal wall; e) 2.0- penetration through the full length of gingival wall and occlusal wall. A similar scoring system was applied for the microleakage starting from the occlusal margin.

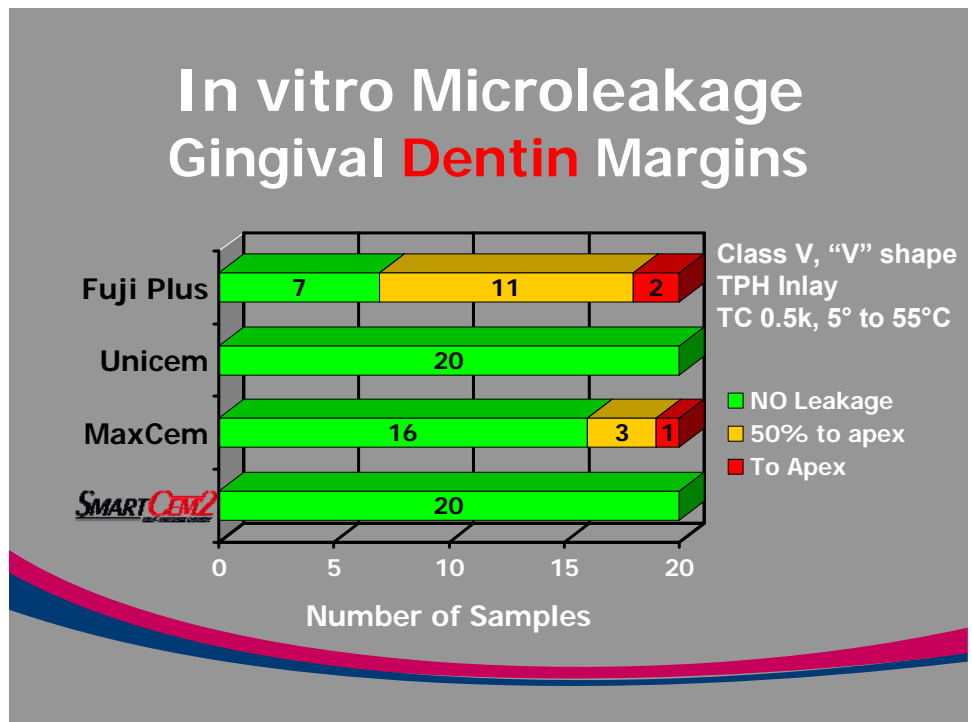
5.3.1. Microleakage in Class V Cavities; Enamel

Figure 7.
Microleakage on extracted human teeth, "v" shaped Class V cavities in both dentin and enamel. Specimens were restored and after 24 hours in water, thermal cycled.



5.3.2. Microleakage in Class V Cavities; Dentin

Figure 8.
Microleakage on extracted human teeth, "v" shaped Class V cavities in both dentin and enamel. Specimens were restored and after 24 hours in water, thermal cycled.



5.4. Shelf-Life & Stability

5.4.1. Chemistry of the Initiator Systems

During the development phase of the SmartCem™2, it was recognized that the traditional components used for the initiation of curing would not be suitable for a self-etching, self-adhesive product. Since it was desired to have a dual curing mechanism in SmartCem™2, the formulation would require dual initiator systems. Thus, due to the use of acidic components within the formulations of SmartCem™2, both the visible light and self-cure initiators would be affected. Both initiator systems utilize basic components (e.g. amines), and these components would be expected to react with the acidic adhesion promoter. This would result in compromised storage stability if the components were allowed to come in contact with each other in the package. A further complication involved the interaction of the acid components with the traditional benzoyl peroxide (BPO) self-cure initiator. This interaction would also lead to degradation of the peroxide initiator over time if allowed to be in contact with the acidic components during storage. Thus, the formulation of the SmartCem™2 required a fresh look at the components of the dual curing composition.

5.4.2. Setting Mechanisms

Based on research findings from the development of Xeno® IV Dual Cure Self-Etching Dental Adhesive, the new, proprietary acid resistant amine was incorporated as part of the visible light initiator system in SmartCem™2. This amine is not reactive with the acidic components of the SmartCem™2, which makes the composition stable to storage conditions, insuring that the visible light activation of SmartCem™2 will occur predictably during the lifetime of the product.

A second research problem involving the self-curing system within the SmartCem™2 provided a novel, proprietary initiating system that also resulted in a shelf-stable material. In this case, the traditional benzoyl peroxide/amine system was replaced with a hydroperoxide/non-amine system.



This new self-cure initiating system has several advantages;

- 1) The new system is insensitive to the acidic components of the SmartCem™2,
- 2) The degradation of the traditional BPO component that leads to changes in working and setting properties is eliminated,
- 3) The degradation of the traditional BPO component with exposure to heat and prolonged storage times is eliminated, and
- 4) The effect of color change of the materials using a BPO/amine self-cure is also eliminated.

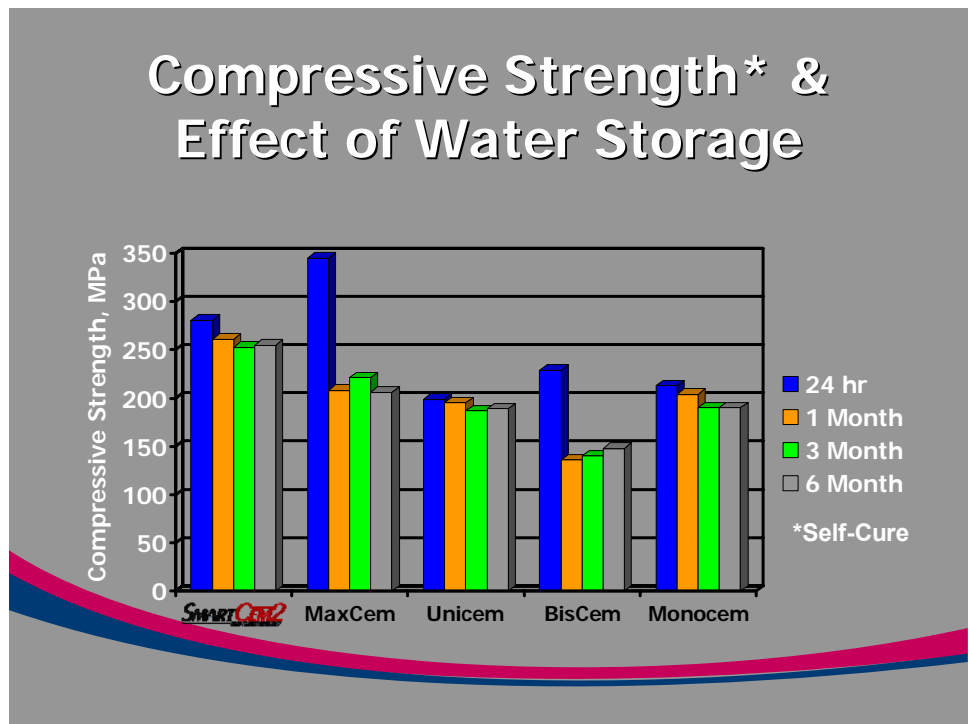
The overall effect of this new self-cure initiator system results in a shelf-stable product with stable and predictable handling properties (e.g. consistent working and setting times), without color changes of the cured SmartCem™2 cement over time. Because the heat sensitive BPO/amine components were replaced, the SmartCem™2 has a significantly improved shelf-life without the need for refrigeration of the product.

6. Other Mechanical, Physical & Miscellaneous Properties

The following sections describe additional mechanical, physical and miscellaneous properties of SmartCem™2. The inherent strength of the SmartCem™2 adds reinforcement to indirect materials, providing enhanced stability and strength of the overall restoration. This obviously is more a factor in all ceramic materials which often require special conditions for bonding. In fact, several manufacturers of ceramic materials caution against the use of the new class of self-adhesive cements with low strength (flexural strength <250 MPa) porcelain. As will be noted from the extensive list of properties that follow, the SmartCem™2 product often demonstrates the most advantageous qualities in each set of test results. The complete descriptions of tests methods are included in the Appendices. Where applicable, standardized test procedures following ISO international standards were employed.

6.1. Compressive Strength

Figure 9.
Compressive strength measured at 24 hours and after storage in water over various period of times.

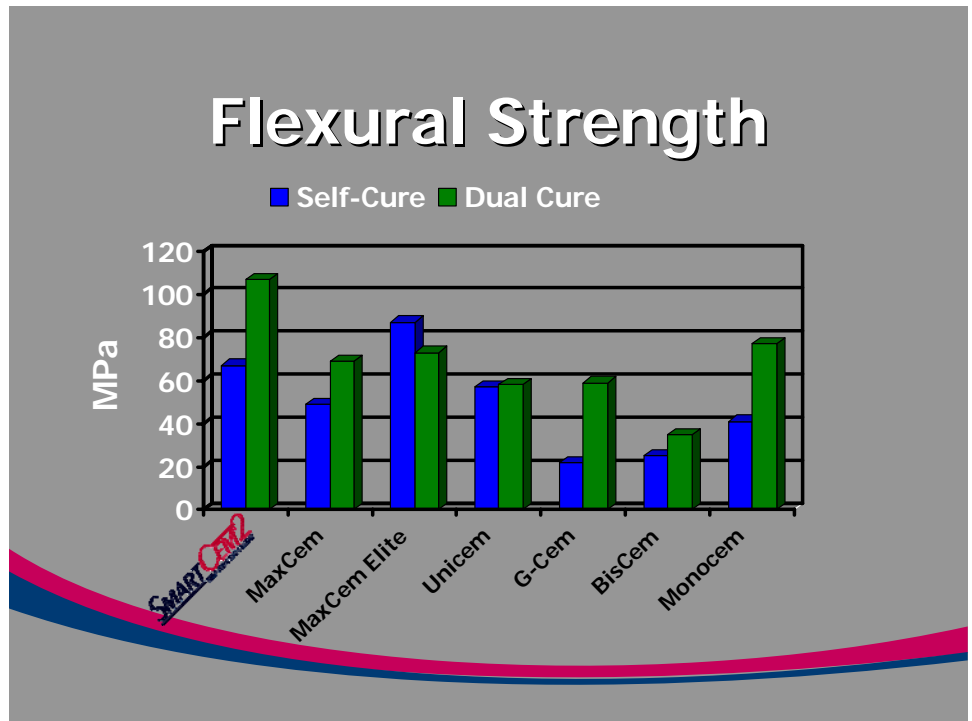


As can be seen, the compressive strength of SmartCem™2 is the highest of the materials tested and remains high even after storage in water for over 6 months. The compressive strength of both MaxCem™ and BisCem, although high as measured at 24 hours, both show a dramatic decrease in the strength after just one month of storage in water. In addition, the

physical appearance of the specimens for these materials shows cracks and crazing on the samples, which suggest some form of degradation of the material could result when placed in contact with fluids in the oral environment.

6.2. Flexural Strength

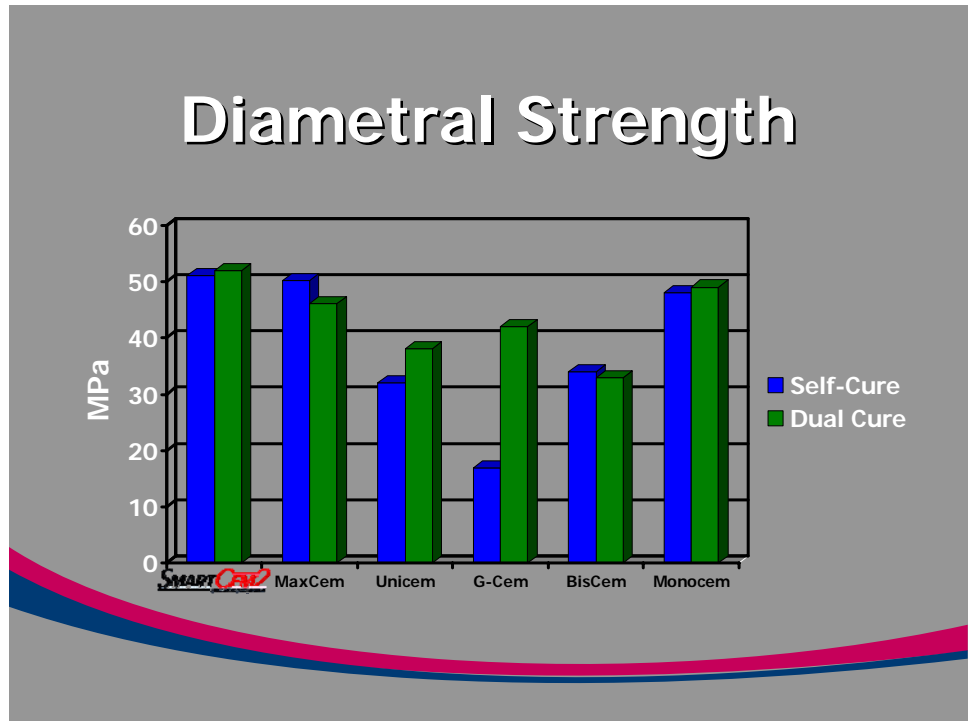
Figure 10.
Flexural strength of self-adhesive cements prepared either using dual cure or self-cure conditions.



SmartCem™2 has exceptionally high flexural strength compared to other self-adhesive resin cements, especially in the dual cure modes.

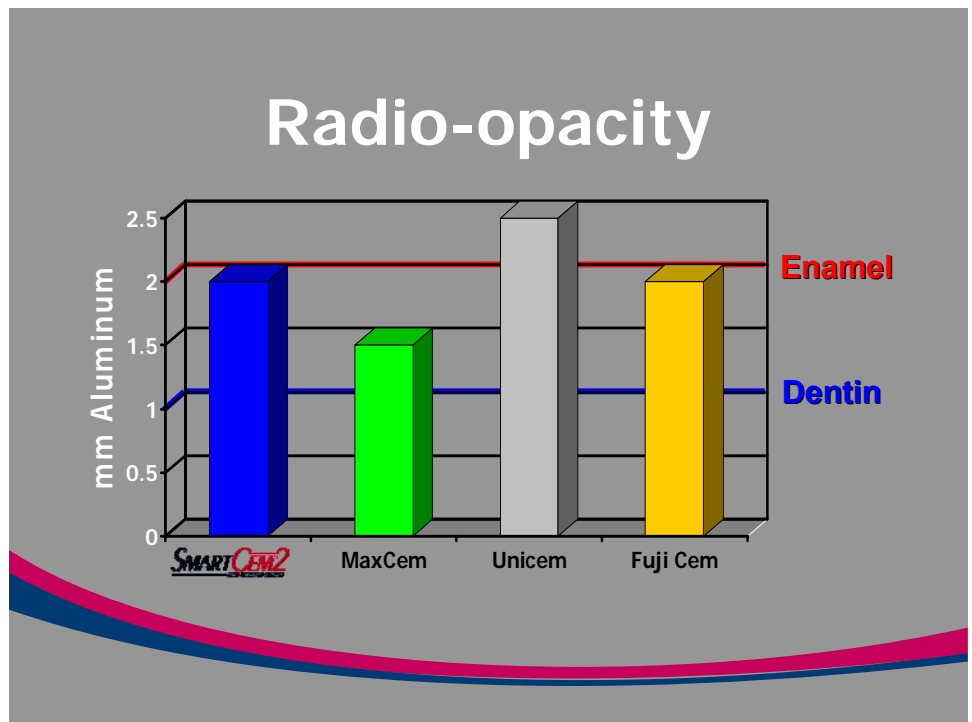
6.3. Diametral Strength

Figure 11.
Diametral strength of self-adhesive cements prepared either using dual cure or self-cure conditions.



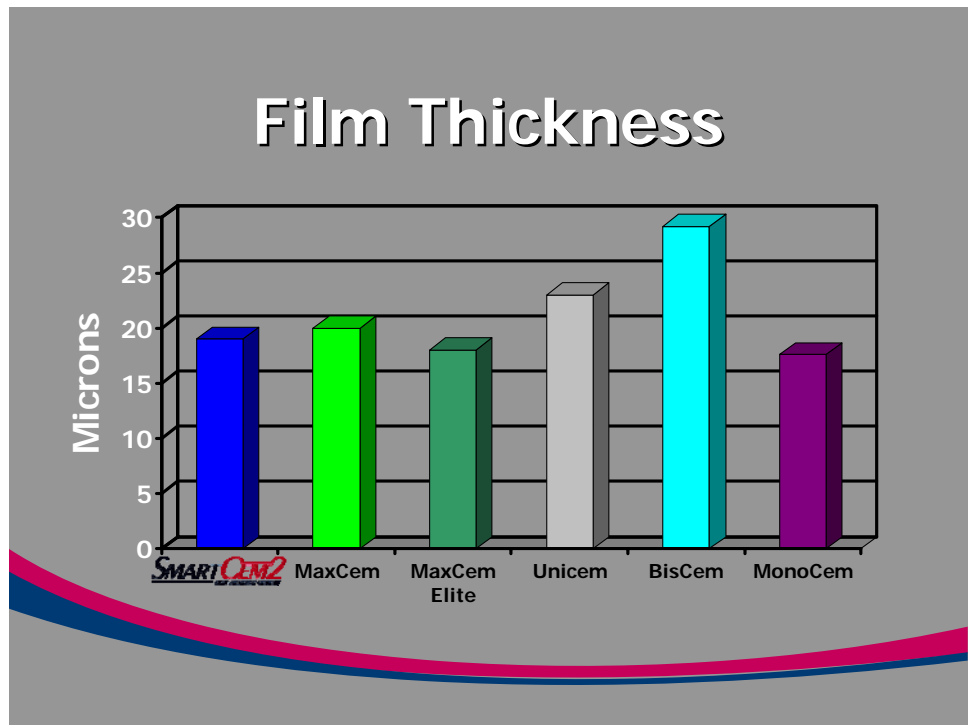
6.4. Radio-Opacity

Figure 12.
The radio-opacity of self-adhesive cements was measured and compared to the approximate radio-opacity of dentin and enamel.



6.5. Film Thickness

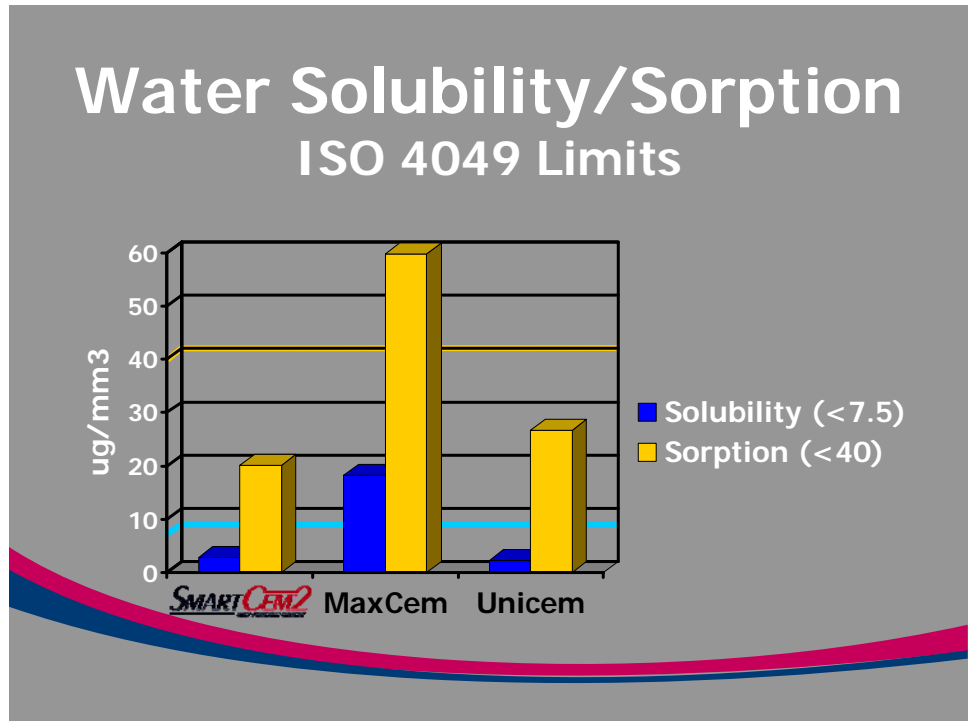
Figure 13.
Cement film thickness as measured in microns via ISO standard test methods.



It is generally accepted that the film thickness of a cement should be 25 microns or less when measured under standard procedures in the laboratory. This will allow for acceptable seating of the indirect restoration. The measured film thickness of BisCem™ exceeded this maximum.

6.6. Water Solubility & Water Sorption

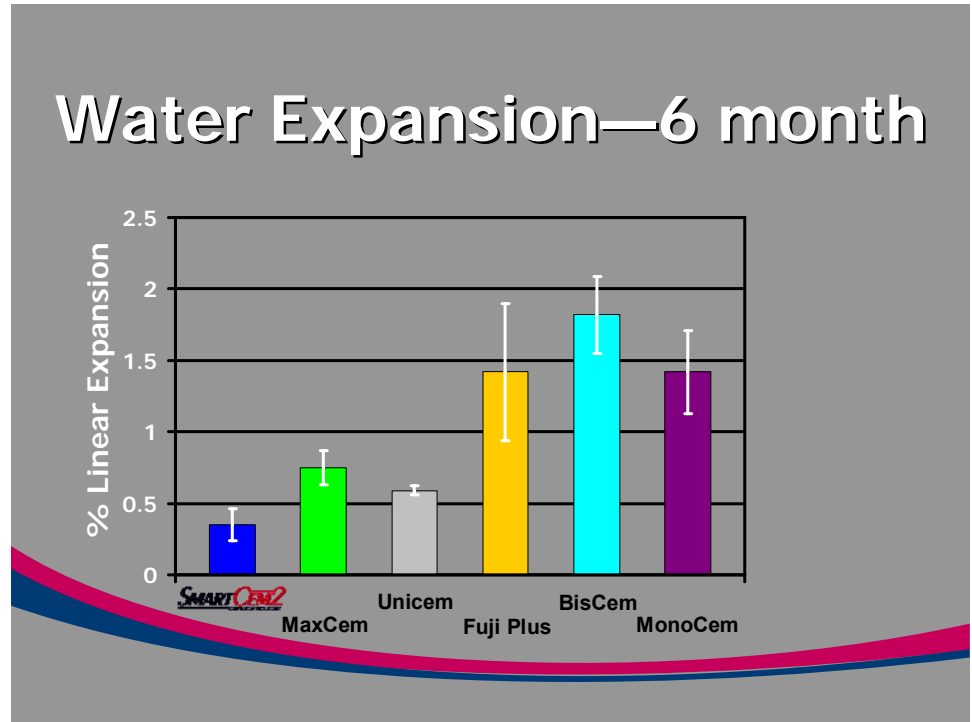
Figure 14.
Water solubility
and water
sorption of self-
adhesive
cements using
ISO standard
test methods.



The water solubility and sorption play an important role in the lifetime of the cement. These important properties are recognized by ISO standards for cements, wherein the limits for water solubility and sorption are specified as maximum values of 7.5 $\mu\text{g}/\text{mm}^3$ and 40 $\mu\text{g}/\text{mm}^3$, respectively. It is noted that MaxCem™ fails both limits in the in vitro testing.

6.7. Water Expansion

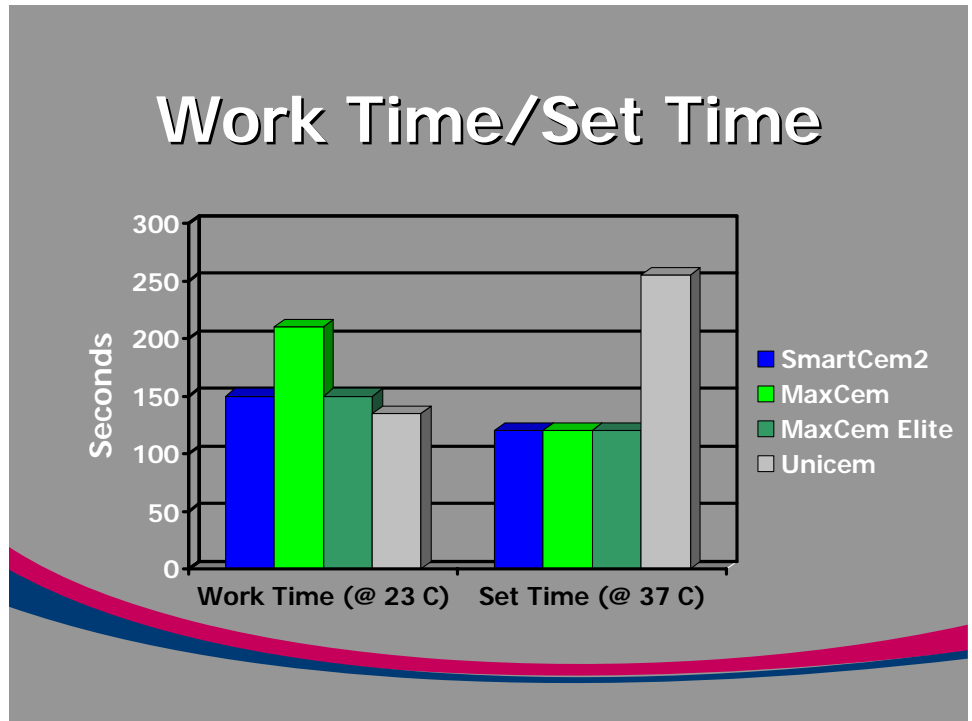
Figure 15.
Water expansion
as measured
over 6 months,
expressed as
linear expansion.



Whether a cement shows expansion is an important criterion for determining the long-term effects of uptake of water on the stability of the restoration. All cements show some degree of expansion when allowed to uptake water over long periods of time, such as would be the case clinically for indirect restorations. However, depending on the class of cements, the water uptake may vary considerably from cement to cement. Even within the class of self-adhesive resins cements included in Figure 15, there is a significant difference among, for example, SmartCem™2 and MaxCem™, BisCem™ or MonoCem™. Generally, an expansion of less than 1% is considered the upper limit for a cement when placing all-ceramic restorations. Thus, as would be expected, Fuji Cem, a glass ionomer material, would only be acceptable for ceramic inlays. The high values measured for the BisCem and MonoCem resin cements noted above may bring into question their suitability for all-ceramic crowns.

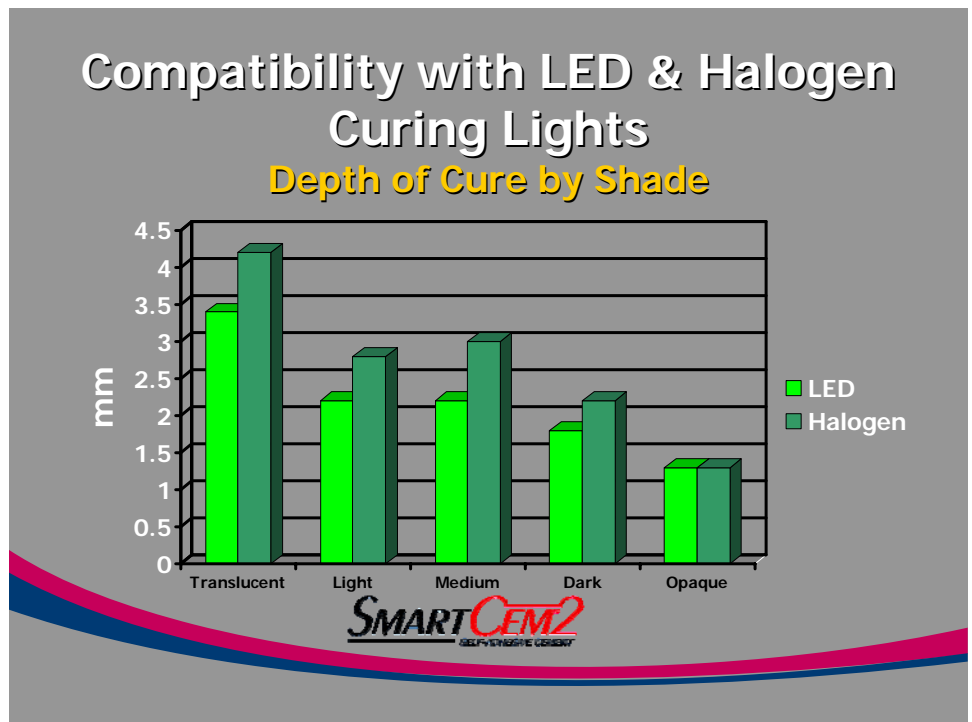
6.8. Working Time & Setting Time

Figure 16.
Work time and set time of cements measured at 23°C and 37°C from start of mix, respectively.



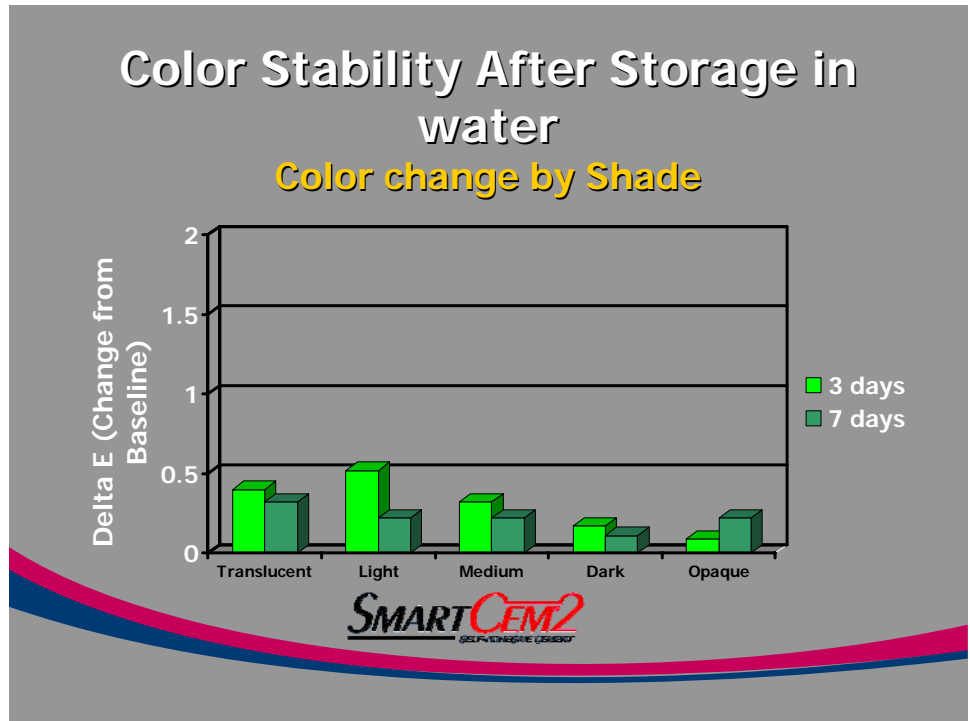
6.9. Compatibility with LED and Halogen Curing Lights

Figure 16.
Depth of Cure of the multiple shades of SmartCem™2 (10 second exposure) using either LED or Halogen curing lights.



6.10. Color Stability

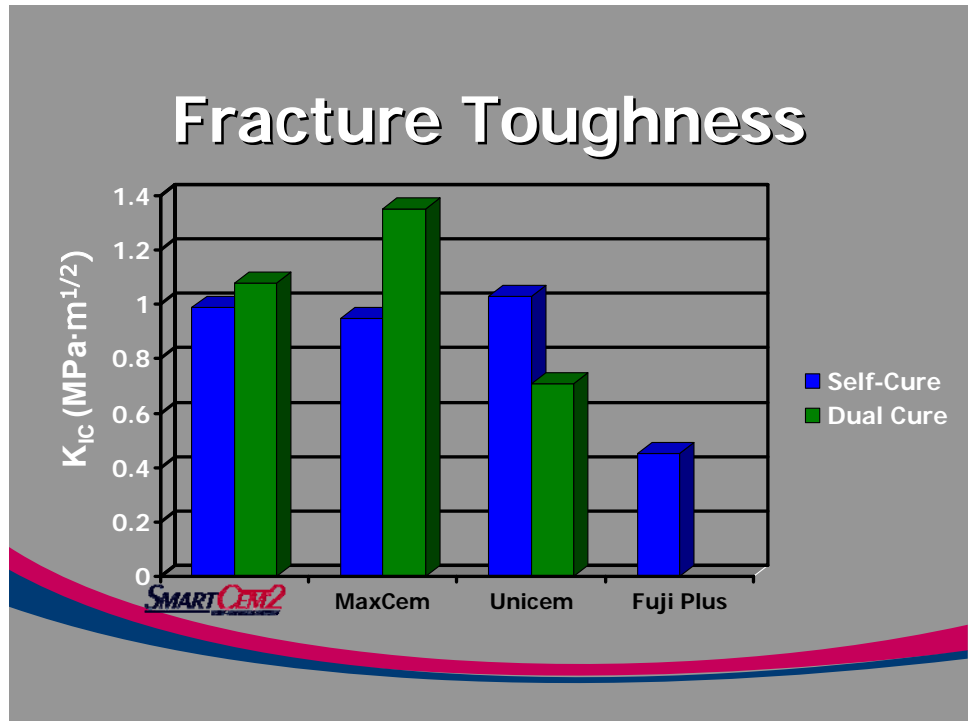
Figure 17.
The color stability of the shades of SmartCemTM2 after exposure to water. Note: No measurable color change was observed under UV irradiation.



Color stability of cements is important for maintaining the esthetic quality of the marginal areas around the restorations. As recorded above, the color stability of SmartCemTM2 is excellent and well below the delta E value of 2.0, the point at which only those with very sharp visual acuity can begin to observe a change in shade. No noticeable color change was observed with all 5 shades of SmartCemTM2 after excessive UV irradiation (see Appendix 9.7.10 for details of the testing protocol).

6.11. Fracture Toughness

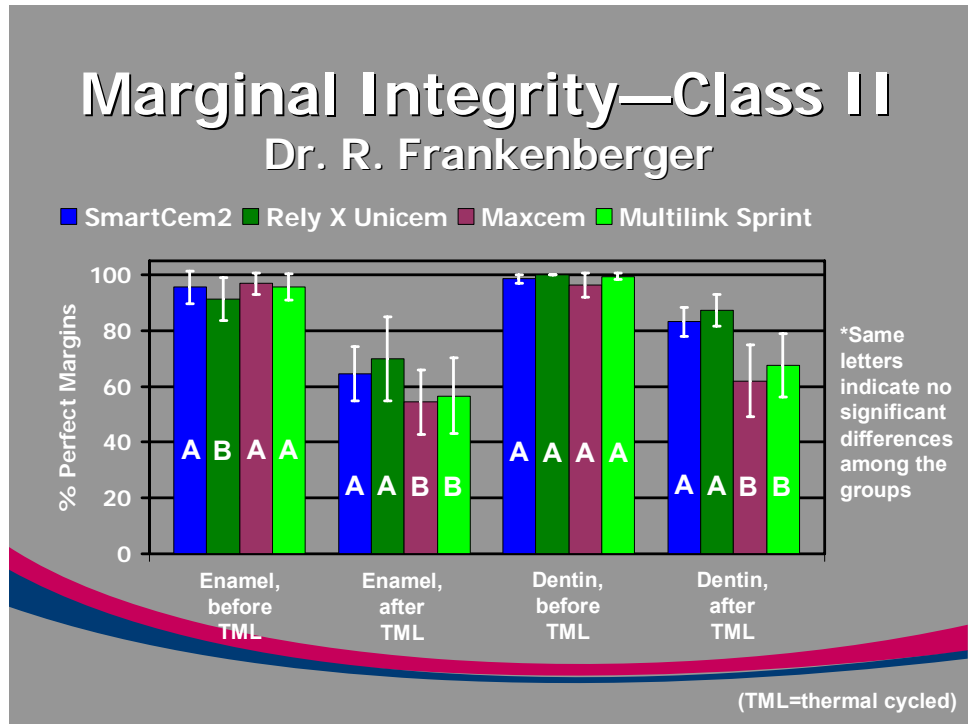
Figure 18.
Fracture toughness of SmartCem™2 and other cements.



The fracture toughness (an approximation of the toughness of a material) is similar among the self-adhesive resin cements and higher than a traditional glass ionomer cement (Fuji™ Plus), as would be expected. Although both the self-cure and dual-cure values are similar for SmartCem™2, there are differences between the self-cure and dual cure values for both MaxCem™ and Rely X™ Unicem.

6.12. Marginal Integrity

Figure 19.
In vitro marginal integrity study of Class II inlays luted with various self-adhesive cements.



The purpose of this study was to evaluate the effectiveness of several self-adhesive cements in providing good marginal integrity of extended MOD ceramic restorations luted in an in vitro clinically relevant test procedure. Therefore, the marginal integrity of the cemented inlays, as measured as percent perfect margins, was evaluated after thermo-mechanical loading of specimens in an artificial oral environment (Details of the testing protocol have been published separately^{2,3}--see also a summary description in Appendix 9.7.12).

Standardized Class II cavity preparations were prepared in human teeth, the treated ceramic inlays were cemented, finished and polished, and stored for 21 days at 37⁰C. After impressions of the restorations were completed, the restored teeth were subjected to thermo-mechanical loading in the artificial oral environment against a steatite antagonist for 100k cycles, while being simultaneously subjected to thermal cycle conditions between 5 and 55⁰C for 2.5k cycles. After the mechanical loading was completed, a new set of impressions were taken of the specimens, from which another epoxy replica was made for comparison to the pre-stress replica. The percent continuous margins were then calculated as marginal



integrity followed by non-parametric statistical analysis for pairwise comparison at the 95% significance level.

After thermal-mechanical loading the results, as shown in Figure 19, indicated that SmartCem™2 exhibited marginal integrity similar to Rely X™ Unicem and significantly better than MaxCem™ and Multilink Sprint.

7. Clinical Evaluations

Introduction:

Clinical trials were conducted on two groups of indirect restorations; those fabricated from lower strength ceramics (IPS Empress) and those fabricated from core reinforced ceramics (Cercon). These two classes of materials were selected because they represent the more challenging clinical applications for adhesive cements. The lower strength ceramics such as IPS Empress are thought by some to require substantial bonding to tooth structure in order to prevent fracture. Core reinforced ceramics such as Cercon can present a challenge with respect to retention because they require a tooth preparation that can exhibit less natural retention vis-à-vis metal and PFM. These trials were considered adequate to support the indications for cementing indirect restorations.

This report provides a summation of the data collected at the six month recall for all three trials.

Clinical Trials:

Clinical trials were initiated at three sites:

Site

Brazil Dental Association

University of Michigan

Eastman Dental Center

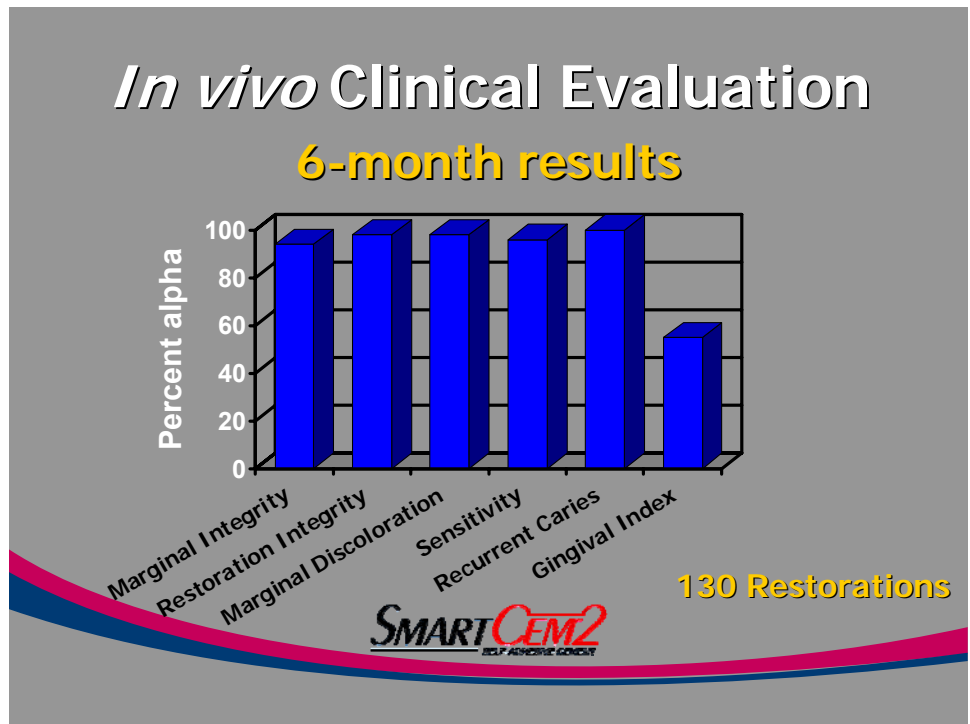
Total Restorations Placed (BL) and Recalled at 6 Months (6m):

	RC Crowns		NRC Crowns		NRC Inlay/Onlay		Total Rest.	
	BL	6M	BL	6M	BL	6M	BL	6M
Test	57	56	51	48	29	26	137	130
RC = Reinforced Ceramic / NRC = Non-Reinforced Ceramic / BL = Baseline								

Results:

A summary of the results of the clinical evaluations is show in Figure 20.

Figure 20.
Summary of 6-month clinical evaluation of 130 ceramic crowns, inlays, and onlays cemented with SmartCem2.



Discussion:

Scoring criteria for all efficacy assessments were designated “alpha” for the best scores, “bravo” for the next best, followed by “charlie” and finally “delta” for the worst. A restoration scoring delta normally indicates a failure.

Marginal integrity is a clinical parameter that monitors the integrity of the junction between restoration and tooth structure over time. It can indicate breakdown of tooth structure, the cement or the restorative material. All restorations were rated alpha at baseline. Ninety-four percent of test restorations maintained this rating at six months. It is not uncommon for marginal integrity scores to shift into lower categories even as early as six months since marginal discrepancies can be created by excess restorative material chipping off within weeks or months following cementation. The bravo ratings (6%) connote identification of

excess cement, not marginal breakdown. Since restorations did not exhibit appreciable marginal breakdown, the performance for this parameter was clinically acceptable.

Restoration integrity is used to record whether a restoration is mobile or missing, in part or in whole. All restorations were rated alpha at baseline. After six months, two test restorations were rated bravo indicating small fractures within the ceramic material. These fractures were not related to the cement and did not require replacement of the restorations. They were likely due to unsupported ceramic, faulty fabrication or hyper-occlusion. One restoration dislodged due to a cement related cause. Therefore the performance for this parameter was clinically acceptable.

Marginal discoloration is a parameter used to determine whether there is leakage occurring at the margins of restoration and tooth structure. One test restoration showed slight discoloration at baseline and three did so after six months (2% bravo). A score of bravo, while observable, is normally considered superficial and not indicative of microleakage. Overall, the incidence of marginal discoloration was low and of only slight severity. Therefore the performance for this parameter was clinically acceptable.

Sensitivity assessed whether teeth became sensitive after the restorative procedure or whether existing sensitivity increased in severity. For the restorations, there was some sensitivity, but most was mild in nature as indicated by the low bravo rating (4%), and mostly pre-existing. Since the cement was not associated with post-operative sensitivity, the performance for this parameter was considered clinically acceptable.

Recurrent caries is scored as absent (alpha) or present (bravo). A positive (bravo) score could mean that caries was not completely removed during tooth preparation or that caries subsequently developed, most likely due to microleakage or macroleakage if there was a significant breach in marginal integrity. There were no incidences of recurrent caries in this trial.

The **gingival index** is a measure of the health status of the marginal gingival tissue surrounding the restoration. The gingival index scores the degree of inflammation with a score of 0 indicating no inflammation. The gingival index was not measured at one study site, however, the investigator did note in his report that the gingiva was in an acceptable state of health after six months. At the baseline evaluation, 52% of test restorations had no gingival inflammation. Most of the restorations evaluated scored a 1, indicating mild inflammation. Mild gingival inflammation is a very common condition, affecting more than 70% of the US population. After six months, there was not an appreciable change in gingival scores and therefore no evidence to suggest that either the restorations or the cements used in these studies had any adverse effects on gingival tissue.

Conclusions:

SmartCem™2 performed within clinically acceptable limits after six months for all clinical parameters evaluated. The cement passed very stringent success criteria for efficacy which allowed no more than one cement related failure in either material class (reinforced and non-reinforced ceramic). There were no reports of adverse device events or unanticipated adverse device effects, nor were there any other clinical observations suggesting a safety concern. Therefore this material is considered safe for use in dental patients.

The manipulative characteristics of this material, including handling, working and set times, excess cement removal and delivery are clinically acceptable as evidenced by favorable commentary by all investigators.

8. Summary

In summary, SmartCem™2 Self-etching Adhesive is suitable for the permanent cementation of metal, PFM, resin/composite, ceramic and porcelain inlays, onlays, crowns and bridges and endodontic posts without application of a separate dentin/enamel adhesive bonding agent/system. As demonstrated through extensive in vitro and in vivo testing, SmartCem™2 Self-Adhesive Cement offers the combination of a high strength self-adhesive cement and convenience in clinical use. Cured SmartCem™2 is essentially hydrophobic, minimizing post-cure water sorption, solubility and hygroscopic expansion. SmartCem™2 is available in a convenient dual-barreled syringe or in the digit® Targeted Delivery System unit dose cartridge to simplify delivery and minimize product waste.

As noted in this technical summary, SmartCem™2 offers numerous features, which include low film thickness, low solubility & expansion, improved stability with consistent work/set times, no refrigeration required, 5 Color-stable Shades in both deliveries, and a fluoride containing glass filler.

9. Appendices: Additional Information

9.1. Appendix A: Directions for Use

SmartCem™2 DIRECTIONS FOR USE – ENGLISH

For dental use only.

USA: Rx only.

1. PRODUCT DESCRIPTION

Caulk Self-Adhesive Cement is a two-component, dual-cure, high strength self-adhesive cement which contains fluoride. Caulk Cement combines esthetic shading with a self-etching adhesive, making it suitable for the permanent cementation of metal, PFM, resin/composite, ceramic and porcelain inlays, onlays, crowns and bridges and endodontic posts without application of a separate dentin/enamel adhesive bonding agent/system. Cured Caulk Cement is essentially hydrophobic, minimizing post-cure water sorption, solubility and hygroscopic expansion.

1.1 Delivery forms

Caulk Cement is available in:

- a convenient dual-barreled syringe
- digit® Targeted Delivery System unit dose cartridge to simplify delivery and minimize product waste
- Caulk Cement is available in 5 shades: translucent, light, medium, dark, and opaque

1.2 Composition

Urethane Dimethacrylate; Di- and Tri-Methacrylate resins; Phosphoric acid modified acrylate resin; Barium Boron FluoroAluminoSilicate Glass; Organic Peroxide Initiator; Camphorquinone (CQ), Photoinitiator; Phosphene Oxide Photoinitiator; Accelerators; Butylated Hydroxy Toluene; UV Stabilizer; Titanium Dioxide; Iron Oxide; Hydrophobic Amorphous Silicon Dioxide

1.3 Indications

Caulk Cement is intended for the cementation of indirect restorations including ceramic, composite and metal-based inlays, onlays, crowns, bridges, and posts.

1.4 Contraindications

1. Caulk Cement is contraindicated for use with patients who have a history of severe allergic reaction to methacrylate resins or any of the components.
2. Caulk Cement is contraindicated for direct application to dental pulp tissue (direct pulp capping).

1.5 Compatible adhesives

Caulk Cement is compatible with all DENTSPLY adhesives designed for use with dual-cured resin based materials. For details, see complete directions for use of the respective adhesive. The use of other dentin and enamel adhesive systems is at the discretion and sole responsibility of the dental practitioner.

2. GENERAL SAFETY NOTES

Be aware of the following general safety notes and the special safety notes in other chapter of these directions for use.

2.1 Warnings

1. Caulk Cement is acidic in nature and contains polymerizable acrylate and methacrylate monomers which may be irritating to skin, eyes and oral mucosa, and may cause allergic contact dermatitis in susceptible persons.

Avoid eye contact to prevent irritation and possible corneal damage. In case of contact with eyes, rinse immediately with plenty of water and seek medical attention.

Avoid skin contact to prevent irritation and possible allergic response. In case of contact, reddish rashes may be seen on the skin. If contact with skin occurs, immediately remove material with cotton and wash thoroughly with water and soap. In case of skin sensitization or rash, discontinue use and seek medical attention.

Avoid contact with oral soft tissues/mucosa to prevent inflammation. If accidental contact occurs, immediately remove material from the tissues. Flush mucosa with plenty of water after the restoration is completed and expectorate/evacuate the water. If sensitization of mucosa persists, seek medical attention.

2.2 Precautions

1. This product is intended to be used only as specifically outlined in the Directions for Use. Any use of this product inconsistent with the Directions for Use is at the discretion and sole responsibility of the practitioner.

2. Wear suitable protective eyewear, clothing and gloves. Protective eyewear is recommended for patients.

3. Syringe should be tightly closed by replacing the original cap immediately after use.

4. Caulk Cement behaves differently intraorally than in ambient operatory conditions. The set of Caulk Cement is accelerated by the warmth and moisture of the oral environment and/or ambient or operatory light. After placing Caulk Cement in contact with tooth structure, e.g., within endodontic post space or in inlay/onlay preparations, immediately seat restoration. Any delay may allow polymerization to begin, which may prevent complete seating of the restoration. Cement will set in the mouth in approximately 3 minutes while it may take more than 6 minutes to set in extraoral, ambient conditions.

5. Caulk Cement should extrude easily. **DO NOT USE EXCESSIVE FORCE.** Excessive pressure may result in unanticipated extrusion of the material or cause syringe rupture.

6. Some porcelain/ceramic manufacturers do not recommend the use of the self-adhesive cement category with posterior all-ceramic restorations fabricated with lower strength ceramics (flexural strength less than 250MPa).

7. In cases of minimally retentive preparations, conventional bonding should be considered.

8. This class of material is not recommended for the cementation of veneers.

9. Interactions:

- Eugenol containing materials should not be used in conjunction with this product because they may interfere with hardening and cause softening of the polymeric components of the material.
- Contact with some astringent solutions may interfere with hardening of the polymeric components of the material.

2.3 Storage

Caulk Cement should be kept out of direct sunlight and stored in a well ventilated place at temperatures between 2°-24°C/35°-75°F. Allow material to reach room temperature prior to use. Protect from moisture. Do not freeze. Do not use after expiration date.

2.4 Adverse reactions

1. Product may irritate the eyes and skin. **Eye contact:** irritation and possible corneal damage. **Skin contact:** irritation or possible allergic response. Reddish rashes may be seen on the skin. **Mucous membranes:** inflammation. (See Warnings)
2. Product may cause pulpal effects. (See Contraindications)

3. STEP-BY-STEP INSTRUCTIONS

3.1 Preparation of the restoration

Metal Restorations

Internal surfaces of restorations should be clean and dry prior to cementation. Internal surface microetching (sandblasting with 50µ alumina) of metal surfaces of the restoration is recommended.

Ceramic/Composite Restorations

Follow the dental laboratory or restoration manufacturer's instructions for pre-treatment, if required. Restorations designed to be silanated or if the internal silanated surface has been disturbed during try-in, apply Calibra® Silane Coupling Agent (available separately) according to the manufacturer's instructions.

3.2 Preparation of the tooth

Remove temporary restoration and excess temporary cement using an explorer, a rubber cup and a prophy paste or water/flour of pumice. Rinse thoroughly and carefully blot dry preparation with a moist cotton pellet. Dentin should be blotted until there is no pooling of water, leaving a moist, glistening surface. Avoid desiccating. Avoid contamination. Etching of tooth surfaces is NOT recommended.

Technique Tip: The adjacent teeth and/or the external surfaces of the restoration may be lubricated with a water soluble medium to ease clean up of excess cement.

3.3 Cementation technique

Danger of injury due to excessive force

- Apply slow and steady pressure on the syringe
- Do not use excessive force – digit® unit dose or dual barrel syringe rupture may result

3.3.1 Dual Barreled syringe dispensed

1. Remove syringe cap. Dispense and discard a small amount of material from the dual-barreled syringe. Be sure material is flowing freely from both ports. Holding syringe vertically, carefully wipe away excess so base and catalyst do not cross contaminate and cause obstruction of the ports. Save syringe cap for replacement following use.
2. Install a mixing tip on the cartridge by lining up the v-shaped notch on the outside of the mix tip with the v-shape notch on the syringe flange. Turn colored mix tip cap 90 degrees in a clockwise direction to lock in place on syringe.
3. Gently depress syringe plungers to begin the flow of material. **DO NOT USE EXCESSIVE FORCE.** If force is encountered, remove syringe from operating field, remove and discard mix tip. Check for obstruction and confirm material is flowing from both syringe barrels. Wipe barrels and install new mix tip as outlined above. Dispense a small amount through the mix tip onto a mixing pad and discard.
4. Without delay, using gentle pressure, apply a thin, uniform layer of cement to the entire internal surface of the restoration directly from the mix tip. At room temperature, Caulk Cement offers a minimum work time of 2 minutes. **Technique Tip:** The mixing tip may be bent slightly to allow direct intraoral access for placement of cement into preparations with internal anatomy. For endodontic post spaces, use of a Lentulo Spiral or metal file to aid placement in the post space is recommended.
5. **Immediately** seat the restoration in the mouth. Verify complete seating. A gentle rocking or vibratory motion may be helpful to insure optimal seating.
6. Following placement, Caulk Cement will self-cure to an initial set in the mouth in approximately 3 minutes. Protect restoration from contamination and movement during the setting time.

OR

3.3.2 digit@ unit dose dispensed

1. Select unit dose cartridge material to be used. Assemble clean syringe by inserting plunger into open end of syringe barrel. Assure that plunger moves freely within syringe barrel. Have clean, assembled syringe, new mix tip and new intra-oral tip (if applicable) available. Do not assemble cartridge/mix tip until ready for use.
2. When ready for use, place the circular disk of the unit dose cartridge into the slotted end on the syringe plunger. Bend unit dose cartridge to snap off the circular disk. (Do not twist cartridge.)
Disc should snap off cleanly, exposing both cartridge ports.
3. Grasp clean mix tip in one hand, and cartridge in the other. Insert unit dose cartridge into the mix tip assembly. Press firmly until the cartridge is fully seated and snapped into place. Both cartridge spurs must be fully locked into mix tip slots before proceeding. If not locked, apply additional pressure until locking, or discard mix tip and select another tip for assembly.
4. If desired for material application, attach intra oral tip (see complete Directions for Use for cartridge material selected).
5. Retract syringe plunger until stop is felt. Do not completely remove/disassemble plunger. Place assembled cartridge/mix tip (with attached intra oral tip, if applicable) through syringe side opening, sliding mix tip through barrel end with cross notches. Push cartridge/mix tip

assembly into place. After loading, firmly pull on the end of the mix tip to make sure the loaded cartridge/mix tip is fully seated in the syringe.

6. Depress plunger until both members fully engage cartridge. Away from the patient field, continue to depress plunger until material flows from both ports into the mix tip. Bleed and discard a small amount of mixed material, then proceed immediately to clinical application. To dispense material, apply a slow, steady pressure to plunger (using palm or thumb, as preferred). Excessive force is not necessary. If resistance is encountered, or if excessive force is required, remove the syringe from the patient field and check for plunger obstructions. If cartridge/mix tip obstruction is suspected, remove cartridge/mix tip assembly and replace, following steps above.

7. Without delay, using gentle pressure, apply a thin, uniform layer of cement to the entire internal surface of the restoration directly from the mix tip. At room temperature, Caulk Cement offers a minimum work time of 2 minutes. **Technique Tip:** The mixing tip may be bent slightly to allow direct intraoral access for placement of cement into preparations with internal anatomy. For endodontic post spaces, use of a Lentulo Spiral or metal file to aid placement in the post space is recommended.

8. **Immediately** seat the restoration in the mouth. Verify complete seating. A gentle rocking or vibratory motion may be helpful to insure optimal seating.

9. Following placement, Caulk Cement will self-cure to an initial set in the mouth in approximately 3 minutes. Protect restoration from contamination and movement during the setting time.

10. Allow material to set completely before attempting disassembly. To disassemble, withdraw the plunger, straighten mix tip if bent, and tap the mix tip against the counter to dislodge the cartridge/mix tip assembly. Properly dispose used cartridge/mix tip assembly.

11. Prepare the digit® syringe for subsequent reuse by following instructions below for cleaning and sterilization.

3.4 Cleaning marginal excess Caulk Cement

3.4.1 Self-Cure Cleanup

The excess cement will reach the “gelled” state after approximately 1-2 minutes in the mouth, allowing easy removal. Excess cement will remain in the “gelled” state for approximately 1 minute. If exposed to directed operatory light, “gel” state may be reached sooner and/or remain “gelled” for a shorter period. Immediately after reaching the “gelled” state, floss interproximally to remove excess cement. Complete excess cement removal using an instrument such as a rubber tip, a scaler or an explorer.

3.4.2 Optional Dual-Cure Cleanup

Due to the dual-cure property of Caulk Cement, the operator has the option of utilizing a curing light to facilitate cleanup. Excess cement cleanup may begin immediately following a brief exposure with the curing light. Conventional quartz tungsten halogen or LED lights producing light of wavelength 470nm are recommended. Use of narrow spectrum output lights or high power output may produce unexpected results.

Immediately after seating is verified, briefly light-cure excess cement at the margins by constantly moving the curing light tip around the margins for no more than 10 seconds.

Excess cement will reach a “gelled” state after this 10 seconds cure. **Light curing mixed cement continuously for more than 10 seconds, at any time, will cause cement to adhesively set, making cleanup difficult.**

The excess cement will remain in the “gelled” state for approximately 45 seconds following light exposure. All excess cement must be removed before final self-cure set is achieved, as outlined above. Following all excess removal, exposed margins may be light cured 20-40 seconds to assist restoration stabilization.

3.5 Curing, finishing and dismissal

For metallic, thick or heavily opaqued ceramic or composite, or restorations that otherwise impede the transmission of light, once restoration is stabilized, allow Caulk Cement to self-cure without disturbing. Protect restoration from contamination and movement during the setting time. Following the self-cure set of approximately 6 minutes from start of mix, check and adjust occlusion and polish as necessary. Patient may then be dismissed.

For most non-metallic, light-transmissible ceramic or composite restorations, Caulk Cement may be visible light cured. Once stabilized, light cure all areas of the restoration using a visible light, curing unit designed to cure CQ initiated methacrylates (spectral output including 470nm), with a minimum output of 550mW/cm² for 10 seconds from each direction – buccal, lingual and occlusal. Following the light-curing, check and adjust occlusion and polish as necessary. Patient may then be dismissed.

Inadequate polymerization due to insufficient curing

- Check compatibility of curing light
- Check curing cycle
- Check curing output before each procedure

Important Technique Tips:

- When simultaneously cementing multiple single units or bridgework, it is recommended to employ the light-cure cleanup on one or two adjacent units only, allowing other units' excess to self-cure, providing ample cleanup time.
- Light curing to facilitate cleanup must be accomplished within the first minute following intraoral insertion. Light exposure after 1 minute intraoral time may cause excess cement to adhesively harden to completion.
- Clean excess cement from metal instruments immediately as set cement will adhere to the instrument.

4. HYGIENE

4.1 Cleaning

To clean the digit® syringe, the following procedure is recommended. Fully retract the plunger. If the plunger has excess material build-up clean with an alcohol moistened gauze. For dual-barreled syringe, remove used mixing tip and discard appropriately. Replace original syringe cap prior to storing. The digit® syringe and the dual-barreled syringe may be cleaned by scrubbing with a disposable towel soaked with hot water and soap or detergent.

4.2 Disinfection and/or sterilization

Disinfect dual-barreled syringe and the digit® syringe with a hospital-level, tuberculocidal disinfectant solution according to national/local regulations. Iodophors, sodium hypochlorite (5.25%), chlorine dioxide and dual or synergized quaternary ammoniums are approved disinfectants. Some phenolic-based agents and iodophor-based products may cause surface staining. The disinfectant manufacturer's directions should be followed properly for optimum results. Water-based disinfectant solutions are preferred. **NOTE:** As with any plastic instrument, the digit® syringe may weaken over time.

Following cleaning and disinfection as outlined above, digit® delivery system syringe may be steam autoclaved following autoclave manufacturer's recommendations.

To reassemble the digit® syringe, insert plunger into syringe barrel, and press components together. Prior to each use check to make sure that the digit® syringe plunger is fully engaged and in good working order.

Cross-contamination

- Do not clean, disinfect or reuse digit® unit dose cartridge
- Properly dispose of the used and/or contaminated digit® cartridges and expired cartridges and syringes in accordance with local regulations

5. LOT NUMBER AND EXPIRATION DATE

1. Do not use after expiration date. ISO standard uses: "YYYY/MM."
2. The following numbers should be quoted in all correspondences:
 - Reorder Number
 - Lot Number
 - Expiration Date

Manufactured by:

DENTSPLY Caulk
38 West Clarke Avenue
Milford, DE 19963
Tel.: 1-302-422-4511
www.dentsply.com

Distributed by:

DENTSPLY Canada
161 Vinyl Court
Woodbridge, Ontario
L4L 4A3 Canada

EC REP
DENTSPLY DETREY GmbH



De-Trey-Str.1
78467 Konstanz
Germany
Tel.: 49-7531-583-0
www.dentsply.de
Form #569001 (3/12/08)

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9.2. Appendix B: Shear Bond Strength to Dentin & Enamel Test

Method

To determine the bond strength of the self-adhesive cement(s) (SAC) on tooth structure, extracted human molars were wet ground to expose flat surfaces using 320 and 600 grit abrasive paper. For SAC in the self-cure (SC) mode, the mixed cement was bulk loaded to plastic straw, placed onto tooth surface and allowed self-cure. For SAC in the dual-cure (DC) mode, a thin layer of the mixed material was applied onto preformed and sanded composite rods (TPH™ 3) and placed onto the tooth surface. After 30 seconds, the SAC was light cured for 20 seconds three times along the interface circumference with a Spectrum® 800 Curing Unit set to 550 mw/cm². Specimens (n=5) were stored in 37°C water for 24-hr. Shear bond strength (SBS) was obtained using an Instron 4400 at crosshead speed of 1 mm/min.

9.3. Appendix C: Shear Bond Strengths to Various Substrates

1. *Testing Shear Bond Strength of Self-adhesive Cements to Cured Composite Substrates*

Cured Cristobal Plus discs were conditioned using the following method: the surface was sandblasted with 50 μm Al_2O_3 particles for 10 seconds with Micro Etcher at a distance of 10 mm with a 90° angle of the nozzle to the surface. The disc was rinsed with tap water and ultrasonically cleaned in distilled water for 10 minutes and dried with compressed air for 20 seconds. A plastic straw with a diameter of 3.654 mm was filled with mixed cement and positioned onto the sandblasted composite surface. The flash was gently removed using a dental explorer and the resin cement was allowed to self-cure. After storage in 37°C deionized water for 24 hr, the bond strength was obtained in compressive shear mode using an Instron at a crosshead speed of 1 mm/min.

2. *Testing Shear Bond Strength of Self-adhesive Cements to metals*

The surface of Duceranium U, a base metal alloy, or SMG-CF, a noble metal alloy, both from Dentsply/Ceramco, was conditioned as follows: The surface was sandblasted with 50 μm Al_2O_3 particles for 60 seconds using a Micro Etcher at a distance of 10 mm with a 90° angle of the nozzle to the surface. The specimen was rinsed with tap water and ultrasonically cleaned in distilled water for 10 minutes and dried with compressed air for 20 seconds. A plastic straw with a diameter of 3.654 mm was filled with mixed cement and positioned onto the sandblasted composite surface. The flash was gently removed using a dental explorer and the resin cement was allowed to self-cure. After storage in 37°C deionized water for 24 hr, the bond strength was obtained in compressive shear mode using an Instron at a crosshead speed of 1 mm/min.

3. *Testing Shear Bond Strength of Indirect Resin Cement to a pressable glass ceramic with Dual-Cure Bonding Systems*

The surface of Finesse (Dentsply/Ceramco) was conditioned as follows: An 8% solution of HF was applied to the surface for 2 min., then washed thoroughly for 1 min. under tap water and dried with compressed air for 20". The treated surface was then treated with Caulk Silane Coupling Agent for 1 min. and dried again with compressed air for 20". A plastic straw with a diameter of 3.654 mm was filled with mixed cement and positioned onto the sandblasted composite surface. The flash was gently removed using a dental explorer and the resin cement was allowed to self-cure. After storage in 37⁰C deionized water for 24 hr, the bond strength was obtained in compressive shear mode using an Instron at a crosshead speed of 1 mm/min.

4. *Testing Shear Bond Strength of Indirect Resin Cement to a Zirconia ceramic with Dual-Cure Bonding Systems*

The surface was sandblasted with 50 μm Al₂O₃ particles for 60 seconds using a Micro Etcher at a distance of 10 mm with a 90° angle of the nozzle to the surface. The specimen was rinsed with tap water and ultrasonically cleaned in distilled water for 10 minutes and dried with compressed air for 20 seconds. A plastic straw with a diameter of 3.654 mm was filled with mixed cement and positioned onto the sandblasted composite surface. The flash was gently removed using a dental explorer and the resin cement was allowed to self-cure. After storage in 37⁰C deionized water for 24 hr, the bond strength was obtained in compressive shear mode using an Instron at a crosshead speed of 1 mm/min.

9.4. Appendix D: Crown Retention, Dr. Carlos Muñoz

Carlos Muñoz, DDS, MSD
Professor and Chair
University at Buffalo
School of Dental Medicine
Department of Restorative Dentistry

**Effect of Tooth Preparation Design on the Retention of Crowns
Cemented with a New Self-Adhesive Cement**

Purpose:

The purpose of this study was to evaluate the retention of crowns cemented with a new self-adhesive cement.

Natural tooth preparations with a taper of 10 and 30 degree of Total Occlusal Convergence (TOC) 4 mm in height were prepared. Eighty restorations with each taper were made. Metal crowns made of a base metal alloy were fabricated and luted with the following resin adhesive cements:

1. Caulk Experimental prototype (R0917)	LOT: HL6-91-1T	EXP: 07-2007
2. Kerr's MaxCem	LOT: 44904	EXP: 10-2007
3. 3M's Unicem	LOT: 240592	EXP: 09-2007
4. GC's Fuji Plus	LOT: 0509101	EXP: 09-2007

The cemented crowns were tested using a tensile force at: 1) 24 hours after cementation and 2) 1 week stored at 37 deg C @ 100% relative humidity and 500 thermal cycles.

Methods and Materials:

Teeth Selection, Embedding and Preparation:

One hundred and sixty extracted maxillary or mandibular molars of similar dimensions were mounted in acrylic rings with the long axis perpendicular to the base of the ring and embedded in poly-methyl-methacrylate. Teeth were stored in deionized water at 4 °C in 100% humidity and 0.1 % chloramine-T. The teeth were centered in the ring with the aid of specially constructed centering device.

The acrylic cylinders with the mounted teeth were placed on three-jaw chuck with the aid of a centering device (to eliminate small errors during mounting) into a machinist's lathe. Using diamond and carbide burs the teeth were prepared until a 10° and a 30° of total occlusal convergence was achieved. The margins of the preparations were left with a 0.8 mm internal rounded shoulder. The preparations were standardized to a height of 4.0 mm and 6.0 mm occlusal diameter. After preparation the teeth were stored in 100% humidity to avoid desiccation.

(Figures A & B).

Figure A(10)

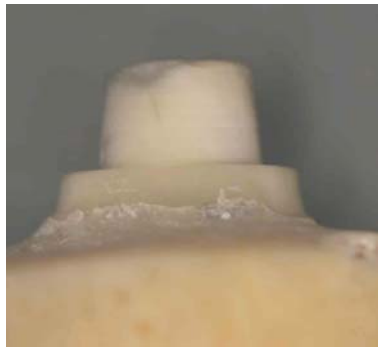
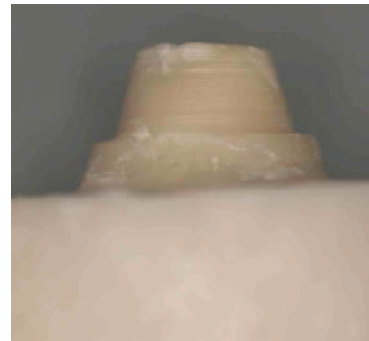


Figure B (30)



Ten teeth were used per group and were prepared to the following specifications:

- 1) Eighty teeth were prepared with a 10 degree TOC (Total Occlusal convergence) and a standardized 4.0 mm of height and 6 mm occlusal diameter.
- 2) Eighty teeth were prepared with a 30 degree TOC and a standardized 4.0 mm of height and 6 mm occlusal diameter.

Of the 80 teeth for each TOC, 20 casting were cemented for each of four resin cements. Ten castings were tested after 24 hours and 10 after aging for one week.

Fabrication of the Castings:

Impressions of all the preparations were made with a poly (vinyl-siloxane) and the impressions poured in die stone (Prima-Rock, WhipMix Corp). 24 hours after pouring, the die stones were removed from the impression and the dies trimmed and sealed. A Stylized crown wax pattern was made for each of the preparations with only one layer of die spacer. An occlusal loop made with a 10 gage wax sprue was made and attached to the occlusal portion of the wax pattern. This loop was used to grip the crown to the jaws of the Instron machine. The stylized wax crown was duplicated in stone and a mold/template was made to fabricate all the other wax pattern crowns.

The wax patterns were marginated, cast on a base-metal alloy (Will-Ceram LiteCast, Ivoclar) and divested. After cleaning the castings, they were examined using a 10X stereo microscope and any nodules removed. The castings were then be fitted to the preparations and the margin fit verified.

Figure C



Cementation and Testing:

The crowns were cleaned with 25 micron aluminum oxide and rinsed with deionized water in an ultrasonic cleaner for 10 minutes.

The cementation of the castings was accomplished following the manufacturers recommended instructions and polymerization times. The casting was seated on the preparation with dynamic pressure and a 15 pound load applied for 5 minutes. After 20 minutes, any excess cement was cleaned and the cemented castings stored in 100% humidity @ 37°C for 24 hours before testing. No external light curing was done to the cements.

Twenty castings were luted for each combination of cement. Ten crowns were tested after 24 hours and 10 castings after one week (aging) storage in relative humidity. The castings that were aged, were first thermal cycled for 500 cycles at 5 and 55°C, 48 hours after cementation using a dwell time of 30 seconds then stored again in 100% humidity @ 37°C until testing.

The retentive bond strength was measured using an MTS Universal Testing machine (1125) at a crosshead speed of 0.1 cm/min. The teeth were mounted in a testing jig, which had a quick chain release fixture/grip that attaches to the loop of the casting

The load required to debond the specimens was recorded in Kg and the mean retentive strength of the ten specimens calculated and reported in mega-pascals using the following equation:

$$\text{Tensile Bond Strength (MPa)} = \text{force (Kg)} / \text{Surface area (cm}^2\text{)} \times 0.09807.$$

The last number was used to convert kg/cm^2 to MPa. The surface area of each specimen was calculated as the sum of the conical surface of the frustum and the surface area of the top of the frustum.

The retention strength for each preparation for the luting cements, aging and TOC were analyzed by using a three-way analysis of variance (ANOVA). All tests were conducted at $\alpha = 0.05$.

Following testing of the specimens, the mode of failure was classified into one of three groups by visual and light microscope inspection. The three groups were:

- A.- Cohesive failure, where the failure occurred either completely within the tooth or completely within the luting agent
- B.- Adhesive failure, where the bond fails between the composite resin and the tooth with no cement left on the tooth and or the crown.
- C.- Fracture of the crown and/or tooth.

Results :

The mean tensile strength of the four luting agents is presented in Table 1 and Figure 1. The three main effects evaluated were TOC (10 and 30), time (24 hours and one week) and cements. A three way ANOVA indicates that there was significant difference among two of the main effects: cements and taper ($p < 0.001$) in regards to the retentive strength and no significant difference for time ($p > 0.660$). (Table 2)

Significant cement by taper interaction was found ($p = 0.030$), significant cement by time interaction ($p = 0.021$) and significant interaction between taper and time ($p < 0.001$). The Tukey multiple comparisons test was used to evaluate the source of the differences at $\alpha = 0.05$.

The mean retentive strength of the Caulk experimental and Unicem were statistically higher than Maxcem and Fuji regardless of the time that the cements were tested $p < 0.001$. (Figure 2). When compared, there was no statistical difference among the Caulk experimental and Unicem ($p = 0.518$) and between Maxcem vs. Fuji $p = 0.779$.

There was an statistical difference among cements when the taper was compared. All cements showed a decrease in retentive strength when the crowns were cemented using a 30 degree taper preparation ($p = 0.030$). (Figure 3).

When the data was collapsed and the taper compared for the two time periods, results indicate that there were statistical significant differences among the 10 and 30 degree tapers. However there was no statistical difference for the 10 degree preparations over time. (Figure 4).

The failure type and the retentive strength within the different luting agents is presented in Table 3. Seventy-six % of the restorations tested after 24 hours presented with adhesive failure, followed by cohesive failure 12.5% and 11.5% where the tooth debonded from the acrylic jig or the tooth fractured. For the crowns tested at one week, 87% of the restorations had an adhesive failure and 11.5 % of the restorations had a cohesive failure and only one restoration had a fracture.

Discussion

The purpose of this study was to evaluate if a new self-adhesive resin cement provided better retention than other commercially available self-adhesive luting agents. It was assumed that by changing the TOC of the preparations and preparing molars with minimum occlusal height, the effect of the bond on the retention of the crowns could be evaluated. It was also assumed that storing the crowns for one week and thermal cycling the restorations would have no effect on the overall retention of the crowns. Overall the preparations had a occluso-cervical to bucco-lingual ratio of 0.4 which is the minimum accepted for molars (Parker 1993).

Results of the study indicated that there was a difference between the retention of the different cements. Maxcem which is a self-adhesive resin cement and Fuji Plus which is a glass ionomer measured similar retention values regardless of the TOC or storage time. The values for these two cements were about 40% lower than those obtained for the Caulk experimental and for Unicem, both self-adhesive resin cements.

Total occlusal convergence had a significant effect on the retentive values of the luting agents. The highest values were measured with the 10 TOC and the lowest values were obtained with the 30 TOC. Geometry of the preparation had a significant effect on the glass ionomer cement, even though is well documented in the literature that glass ionomers form covalent bonds with the tooth structure, but this chemical bond has a limiting factor when the TOC is greater than 10 degrees.

The type of failure was influenced by the TOC of the preparations. A higher number of crowns debonded adhesively for the 10 degree taper than for the 30 degree taper. However, there were enough adhesive and cohesive failures on both types of tapers to indicate that there was enough bond among the self-adhesive cements to mask the effect of the severe TOC, especially between Caulk experimental and Unicem.

Conclusions:

- 1.- Caulk's experimental and Unicem luting agents had the highest retentive strength of all the cements.
- 2.- Maxcem and Fuji-Plus had the lowest retentive strength for both tapers and time.
- 3.- There was a significant difference in strength for crowns cemented with 10 and 30 TOC.

Table 1.- Strength of a Self-Adhesive Cement to Metal Crowns (MPa)

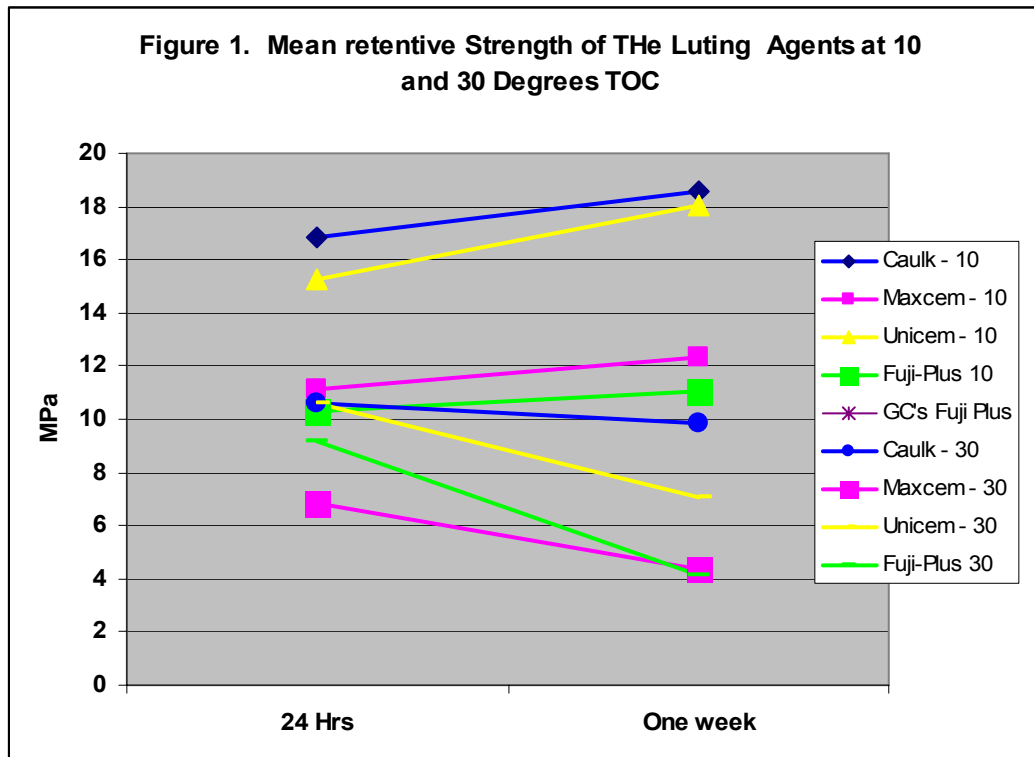
Testing Time	24 Hours		1 Week-Thermal cycled	
	Mean	SD	Mean	SD
10 Degrees TOC				
Caulk Self-adhesive cement	16.86	3.81	18.60	2.12
Kerr's MaxCem	11.14	3.24	12.32	4.03
3M's Unicem	15.27	1.36	18.04	1.59
GC's Fuji Plus	10.30	3.40	11.08	2.10
30 Degrees TOC				
Caulk Self-adhesive cement	10.59	1.30	7.86	1.6
Kerr's MaxCem	6.87	2.6	4.35	1.57
3M's Unicem	10.58	2.4	7.06	1.61
GC's Fuji Plus	9.17	2.1	4.11	1.52

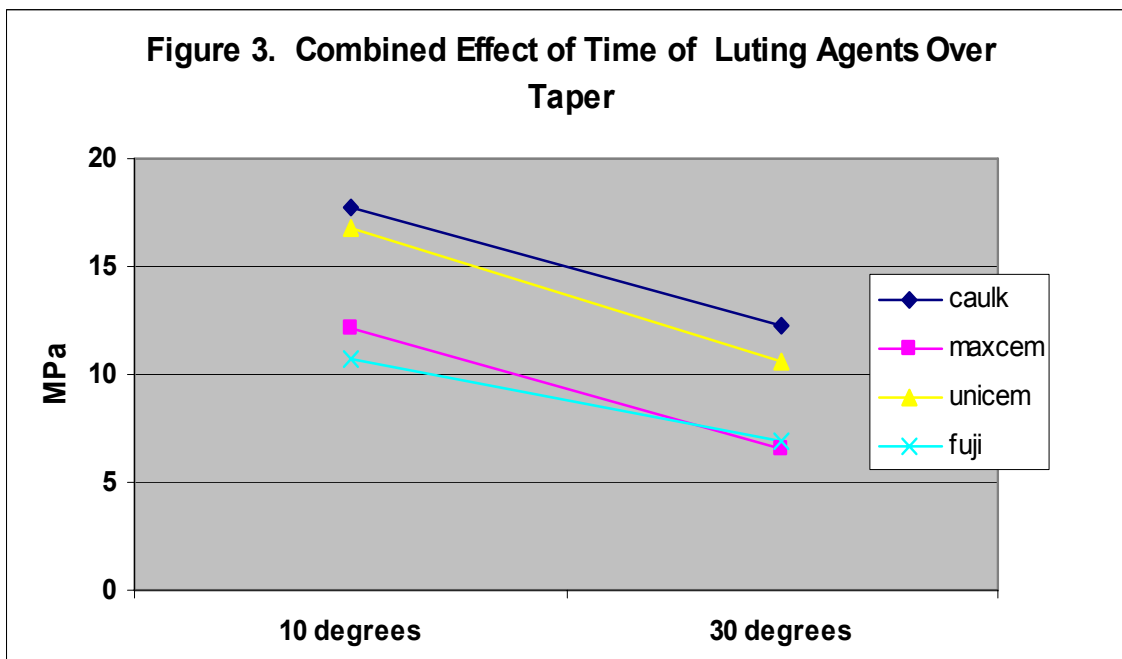
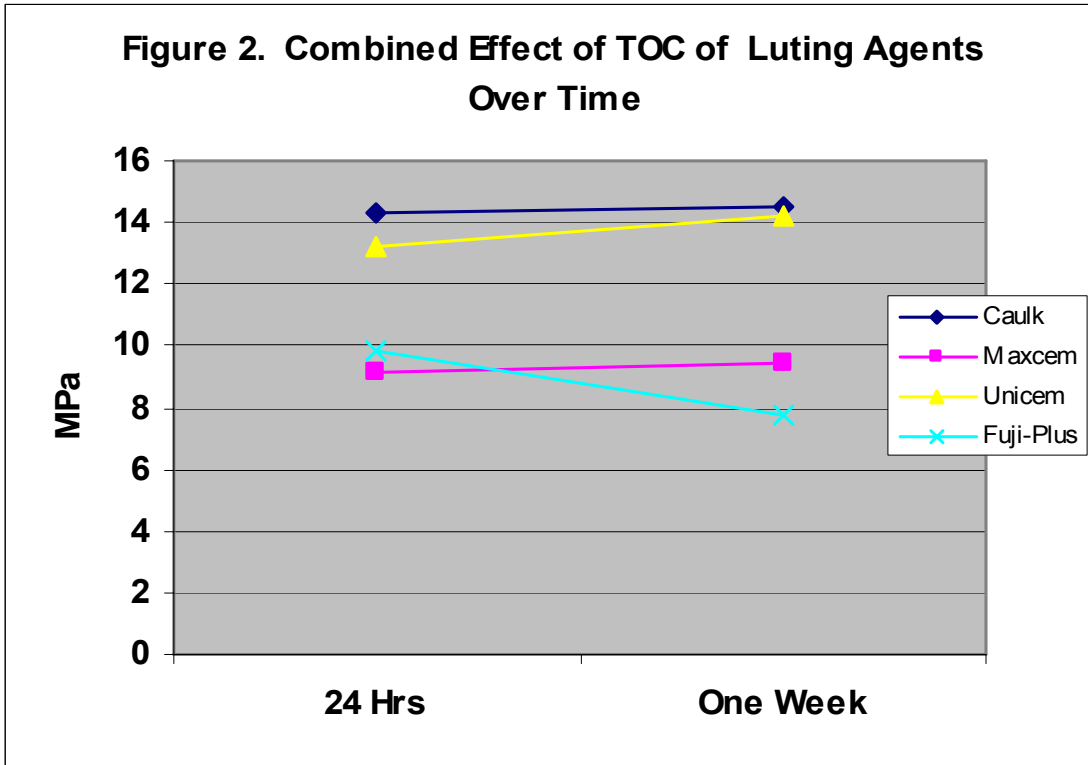
Table 2. Three-way ANOVA for three Main Effects: TOC, Luting Agents and Time

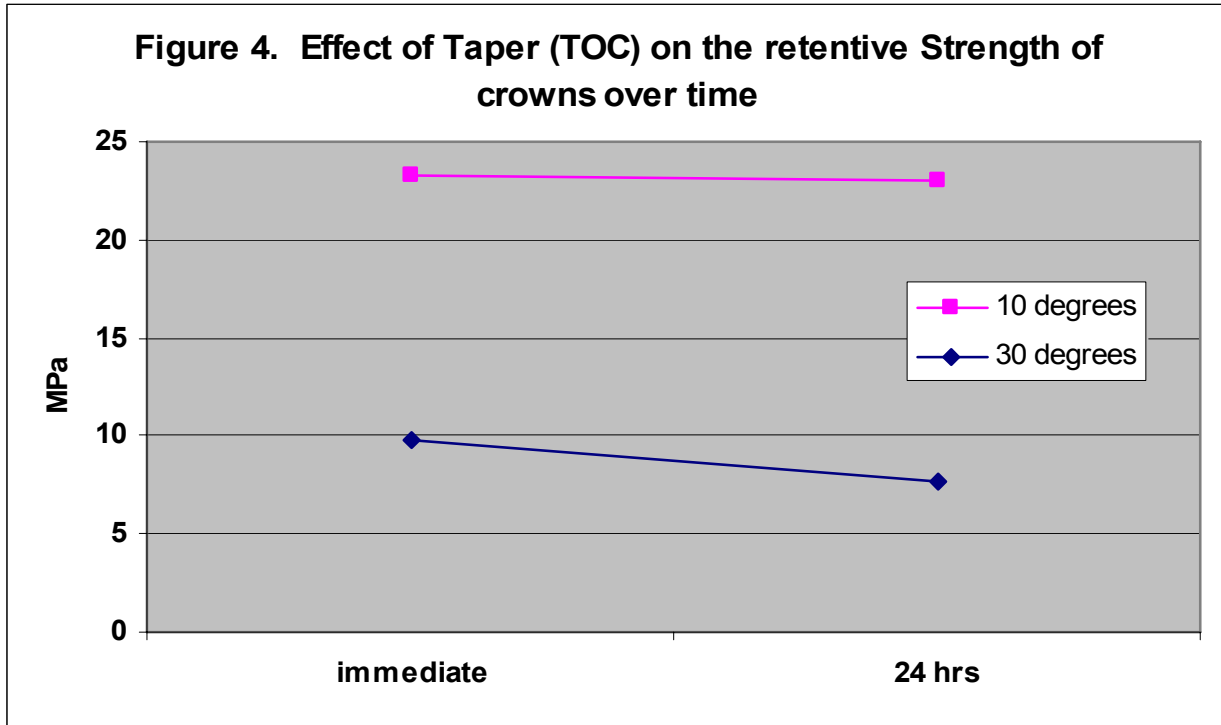
	DF	Sum of Squares	Mean Square	F Value	P Value
cement	3	1009.4	336.5	65.5	<0.001
10 vs. 30 degrees	1	1241.8	1241.8	241.6	<0.001
24 hrs vs. one week	1	0.998	0.998	0.194	0.660
cement x 10 vs. 30 degrees	3	47.3	15.7	3.10	0.030
cement x 24 hrs vs. one week	3	51.5	17.1	3.30	0.021
10 - 30 degrees x 24 hrs vs. one week	1	149.1	149.4	29.00	<0.001
cement x 10 vs. 30 degrees x 24 hours vs. one week	3	22.8	7.6	1.50	0.223
residual	144	739.9	5.139		

Table 3.- Type of failure for each cement group (N = 10)

		10 Degrees (TOC)			30 Degrees (TOC)		
		Adhesive	Cohesive	Fracture	Adhesive	Cohesive	Fracture
24 Hours	Caulk Self-adhesive cement	6	2	2	9	1	1
	Kerr's MaxCem	9	1	0	10	0	0
	3M's Unicem	7	1	2	8	2	0
	GC's Fuji Plus	7	2	1	9	1	0
One Week	Caulk Self-adhesive cement	7	1	2	8	2	0
	Kerr's MaxCem	9	1	0	10	0	0
	3M's Unicem	7	1	2	9	1	0
	GC's Fuji Plus	9	1	0	8	2	0
Total % Failure		76%	12.50%	11.50%	87.50%	11%	2.50%







9.5. Appendix E: Crown Retention, Dr. John Burgess

Retention of Crowns Bonded with four cements

Purpose

To measure retention of crowns cemented with four adhesive cements.

Experimental Design

Cementation Techniques:

Group 1 – Experimental (L. D. Caulk)

Group 2 – Unicem Clicker (3M ESPE)

Group 3 – MaxCem (Kerr)

Group 4 – Unicem Aplicap (3M ESPE)

Replications: 10

Material	Manufacturer	Lot No.	Exp. Date
Experimental	L.D. Caulk	HL6-91-1T	2007/07
MaxCem	Kerr	445795	2007/10
Unicem Clicker	3M ESPE	275043	2008/07
Unicem Aplicap	3M ESPE	250890	2007/12

Materials and Methods

Extracted teeth with notched roots were retained in cylinders filled with acrylic resin. The occlusal surfaces were ground flat and placed into a lathe for precise uniform reduction with diamond cutting tools to produce a uniform crown preparation with exact taper, diameter and fit. An orientation groove was placed into the occlusal surface of preparation by hand using a 69 L bur and high-speed hand piece.



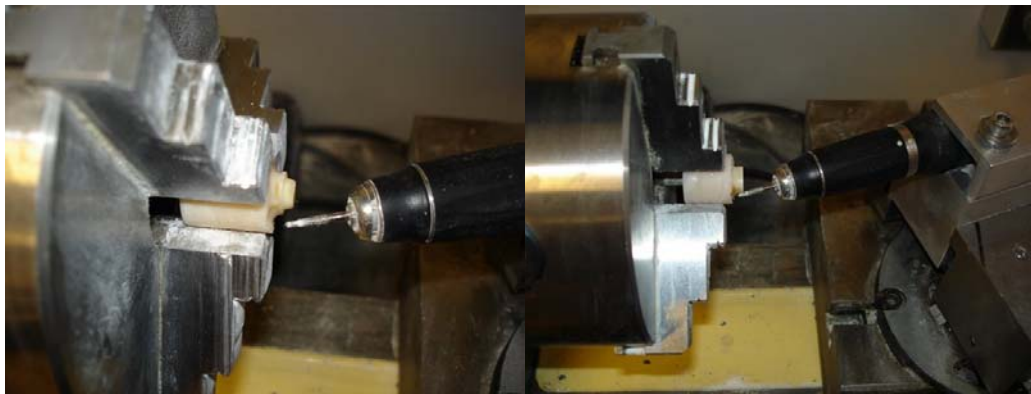
Tooth selection – Notch Preparation



Embedding in acrylic – Preparation of flat surface



Mounted tooth in lathe



Crown preparation

After the teeth have been prepared to uniform dimensions, Zirconia crowns were waxed and milled and a hole is prepared. After sintering, the finished crowns individually fit on the tooth, margins checked for opening and fit (explorer does not catch) and the crowns

cemented. The cements were mixed following the manufacturers directions and a 2kg weight was placed on the cemented crown until the cement had set.



Finished crown

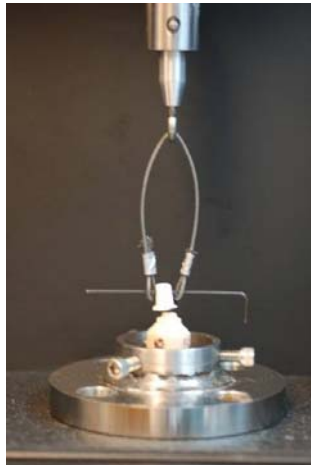
Specimens of Group 1 were cemented with the experimental adhesive cement according to manufacturer's directions.

Specimens of Group 2 were cemented with MaxCem following the manufacturer's directions.

Specimens of Group 3 were cemented with Unicem Clicker according to manufacturer's directions.

Specimens of Group 4 were cemented with Unicem Aplicap according to manufacturer's directions.

Excess cement was carefully removed and the crowns were allowed to set in tap water for 24 hours before debonding. A metal rod was placed through the hole on the crown and through the loops of a wire. Specimens were attached to the hook of the testing machine using the wire (INSTRON Model no: 5565) and loaded in tension at a cross-head speed of 0.5 mm/min until debonding occurred. The force (N) of debonding was recorded. Examination of the failure site was made optically with loops and recorded as cohesive, mixed or adhesive.



Testing of
the
specimen

Results

Materials	Failure Type (# of samples)		
	Adhesive	Cohesive	Mixed
Experimental	3	2	5
Maxcem	9	-	1
Unicem Clicker	10	-	-
Unicem Aplicap	-	-	10

Adhesive failures = failure at the tooth cement interface- tooth was clean

Cohesive failures= failure through the cement with cement on the tooth and on the ceramic.

Mixed failure is a combination of both failure types.

Materials	Failure Load (N) (\pm SD)
Experimental	282.7 (\pm 51)
Maxcem	103.3 (\pm 70)
Unicem Clicker	401.5 (\pm 114)
Unicem Aplicap	493.4 (\pm 161)

The results were analyzed with ANOVA and Tukey B post hoc test. Three significantly different groups were present. MaxCem produced significantly lower tensile strengths than any other cement. The experimental material was intermediate and the Unicem produced the highest tensile strengths. Unicem Clicker and capsule was not significantly different from one another.

Remarks the MaxCem produced lower strengths. Upon closer examination it was noted that the short mixing tips provided with this system did not adequately mix the cement and the cement often did not polymerize.

Test Graphs

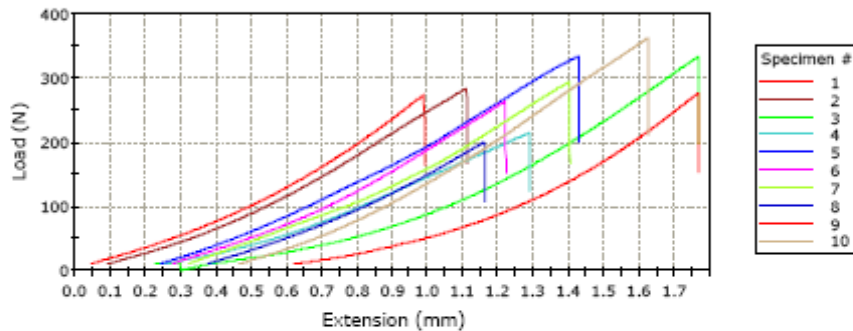
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Dr. John O. Burgess

Wednesday, April 11, 2007

Rate 1	0.50000 mm/min
Specimen label	Experimental Cement
Lot No:	HL6-91-1T
Exp Date:	2007/07
Room T/Humidity	21C/45%

Experimental Cement



	Maximum Load (N)	Tensile extension at Maximum Load (mm)
1	273.08157	0.99096
2	283.62989	1.06427
3	332.10624	1.67344
4	213.38241	1.14798
5	333.62582	1.24124
6	262.96368	0.98600
7	293.15764	1.11976
8	198.66157	0.83041
9	275.29441	1.38916
10	360.97400	1.19980
Mean	282.68772	1.16430
Standard Deviation	51.28973	0.23630

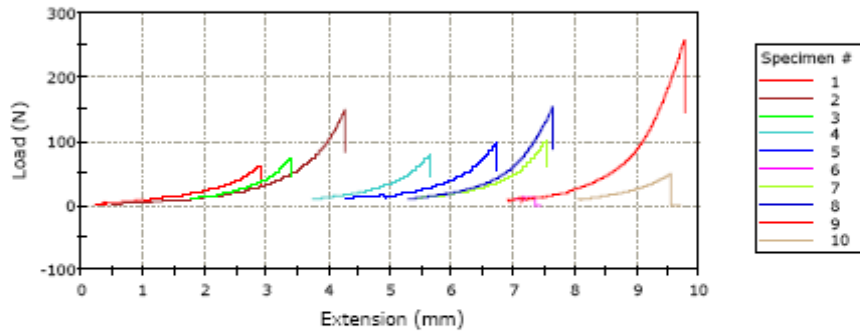
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Dr. John O. Burgess

Friday, March 30, 2007

Rate 1	0.50000 mm/min
Specimen label	Maxcem
Lot No:	445795
Exp Date:	2007/10
Room T/Humidity	23C/43%

Maxcem



	Maximum Load (N)	Tensile extension at Maximum Load (mm)
1	61.69477	2.90252
2	148.94797	4.14577
3	72.75988	3.12541
4	78.41790	5.25491
5	99.11011	6.20561
6	12.78983	6.67146
7	101.62849	6.73273
8	153.81720	6.70145
9	256.61942	8.69400
10	47.52714	8.33345
Mean	103.33127	5.87673
Standard Deviation	68.91897	1.99788

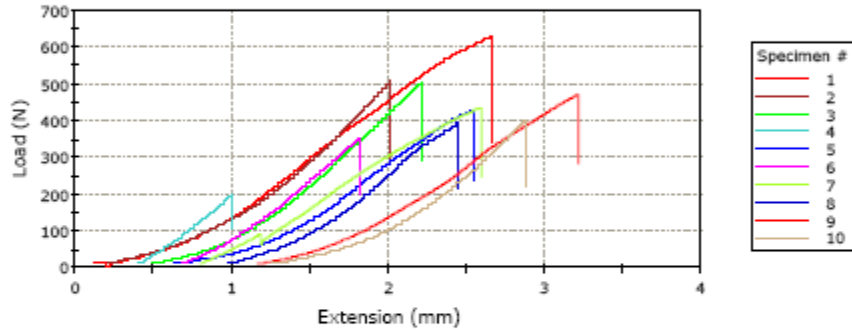
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Dr. John O. Burgess

Tuesday, April 10, 2007

Rate 1	0.50000 mm/min
Specimen label	Unicem Clicker
Lot No:	275043
Exp Date:	2008/07
Room T/Humidity	20C/47%

Unicem Clicker



	Maximum Load (N)	Tensile extension at Maximum Load (mm)
1	629.00543	2.66559
2	505.62311	1.89539
3	503.19549	1.96634
4	194.83294	0.61909
5	429.47717	2.03540
6	352.05927	1.18658
7	435.15045	1.82987
8	392.59460	1.56212
9	471.68236	2.20458
10	400.88397	1.73873
Mean	431.45048	1.77037
Standard Deviation	113.55272	0.56060

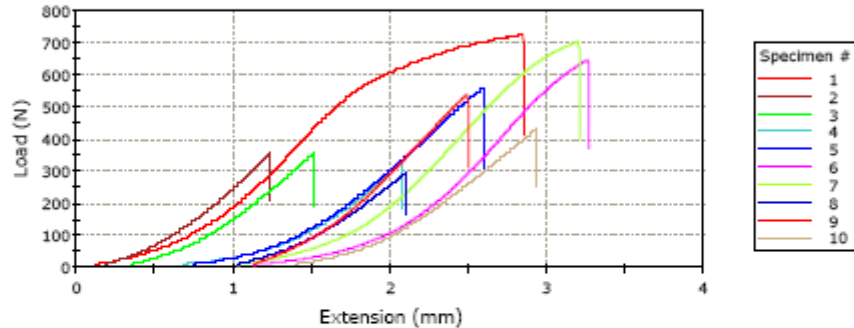
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Dr. John O. Burgess

Friday, September 21, 2007

Rate 1	0.50000 mm/min
Specimen label	Unicem Aplicap
Lot No:	250890
Exp Date:	2007/12
Room T/Humidity	24C/39%

Unicem Aplicap



	Maximum Load (N)	Tensile extension at Maximum Load (mm)
1	722.63137	2.85200
2	355.71527	1.10878
3	355.90570	1.24974
4	329.39598	1.67061
5	558.56135	2.05960
6	646.54663	2.58720
7	702.96891	2.39122
8	294.20147	1.15471
9	537.91042	1.40498
10	429.92037	1.70827
Mean	493.37575	1.81871
Standard Deviation	161.45726	0.62521

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9.6. Appendix F: Microleakage

Non-carious human molars were used in this study. Two class V cavity preparations (V-shape) were made on opposite sides of each non-carious human molar with the occlusal margins in enamel and gingival margins in cementum. Approximate dimensions of the preparations were 4 mm mesiodistally, and 3 mm occluso-gingivally, and 2 mm pulpal.

TPH3 A3.5 was used to make inlays for each restoration. The inlays were cured using the spectrum 800 curing light for 40 seconds, and they were removed from the restorations. The dental cement was applied to the TPH inlays and was pushed into each cavity. All excess cement was removed with a clean microbrush. After waiting for 30 seconds, the restoration was allowed to light cure twice each for 20 seconds with Spectrum 800 at 550 mw/cm² or self-cure for 15 minutes. The restorations were finished and polished with Pogo Polisher System. Thereafter, the samples were stored in 37°C deionized water for 24 hours, followed by thermal cycling (540 cycles between 55°C and 5°C, with dwell time approximately 1 minute in hot and cold bath, and transfer time of 7 seconds in air between baths). Following the thermal cycling period, the teeth were evaluated for marginal microleakage with silver nitrate staining technique. Dental compound was placed in the intra-radicular areas, and the teeth were coated with fingernail polish up to within 2 mm of the restoration to prevent silver nitrate penetration into the teeth from areas other than the cavity preparation. The teeth were placed in 50% (by weight) silver nitrate aqueous solution, and stored in total darkness for 2 hr. The teeth were removed from the silver nitrate solution and rinsed in tap water. After rinsing, the teeth were sectioned longitudinally with a diamond blade (Isomet Low-Speed Saw, Buehler) through the center of the restorations.

The development process consisted of exposure of each sectioned specimen to a fluorescent lamp for 1 hr. Areas of silver nitrate penetration (microleakage) turned black due to the light exposure. Leakage along the gingival wall of the cavity was scored according to the following criteria: a) 0- no dye penetration; b) 0.5- dye penetration to one-half of the distance to the apex.; c) 1- leakage to the bottom (apex) of the cavity; d) 1.5- penetration past the apex and

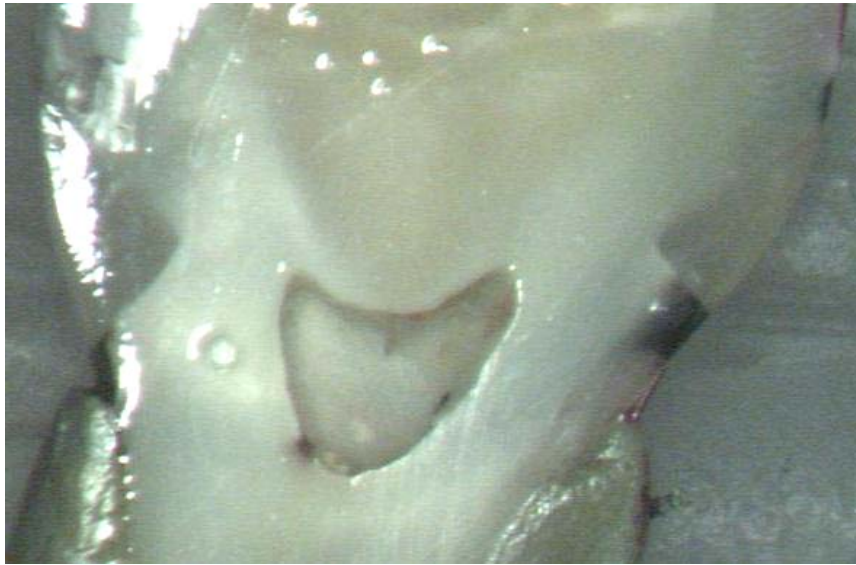
observed on the occlusal wall to the 1/2 the length of the occlusal wall; e) 2.0- penetration through the full length of gingival wall and occlusal wall. A similar scoring system was applied for the microleakage starting from the occlusal margin. In the case of total failure, the score of 2.0 was assigned for microleakage from gingival margin and 1.0 for microleakage from occlusal margin.

Results:

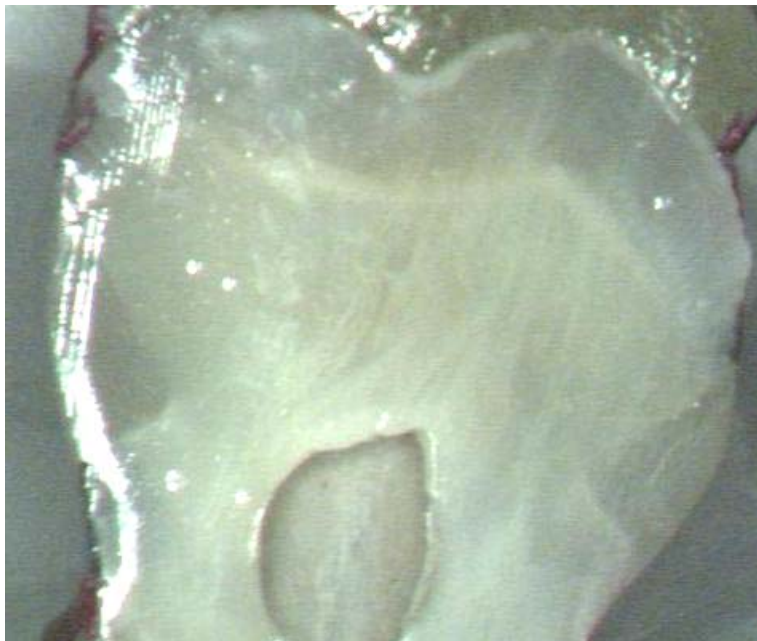
Microleakage of class V cavities restored with TPH3 composite inlays luted with SmartCEM2, Rely-X Unicem Aplicap, Maxcem and Fuji Plus was evaluated with dye penetration method (Table 1 and 2). SmartCEM2, Rely-X Unicem and Maxcem were cured in dual-cure mode while Fuji Plus was self-cured. No microleakage through either gingival or occlusal margin was found with SmartCEM2. Both Rely-X Unicem and Maxcem exhibited minimal but non-zero microleakage. More extensive microleakage was found with Fuji Plus.

Table 1. Microleakage- Penetration through Gingival Margin

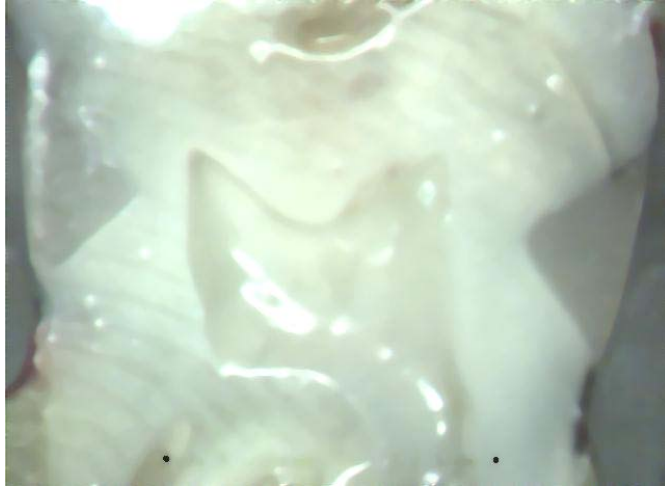
	SmartCEM2	Rely-X Unicem Aplicap	Maxcem	Fuji Plus
Score 0	20	20	16	7
Score 0.5	0	0	3	11
Score 1	0	0	1	2
Score 1.5	0	0	0	0
Score 2	0	0	0	0
Mean	0	0	0.13	0.38
Std.	0	0	0.28	0.32



MaxCem™ sample with microleakage along gingival margin



SmartCem™2 Microleakage Sample



Rely X™ Unicem sample with microleakage along occlusal margin



Fuji Plus Sample with microleakage along gingival and occlusal margins

Table 2. Microleakage- Penetration through Occlusal Margin

	SmartCEM2	Rely-X Unicem Aplicap	Maxcem	Fuji Plus
Score 0	20	16	20	6
Score 0.5	0	4	0	14
Score 1	0	0	0	0
Score 1.5	0	0	0	0
Score 2	0	0	0	0
Mean	0	0.1	0	0.35
Std.	0	0.21	0	0.24

9.7. Appendix G: Other Mechanical, Physical & Miscellaneous Properties

9.7.1. Compressive Strength

Self-adhesive resin cements were selected: experimental automixed paste/paste self-adhesive cement, SmartCem™2 (Dentsply/Caulk), Rely X™ Unicem Aplicap (3M ESPE), MaxCem™ (Kerr), BisCem™ (Bisco) and MonoCem™ (SHOFU). The cylindrical mold (4 mm in diameter and 6 mm in height) was conditioned with a silicone lubricant, placed on a Mylar covered glass plate and slightly overfilled with the mixed cement. The second glass plate was positioned on top of a piece of Mylar covering the mold. The two plates and mold were secured together using a binder clip and the cement was allowed to self-cure for 1 hr at 37 °C. The cylindrical specimens were stored in 37°C deionized water for 24-hr, 1-month, 3-month and 6-month. Compressive strength (CS) was obtained with Instron 4400R at crosshead speed of 5-mm/min.

Results:

Effect of Water Storage on Compressive Strength (MPa)

	SmartCem2	Unicem	Maxcem	MonoCem	BisCem
24 hr	281±20	199±19	345±8	213±29	229±6
1 Month	261±14	195±16	208±28	204±28	136±30
3 Month	253±18	187±31	221±11	190±36	140±26
6 Month	255±15	189±36	206±18	190±23	148±23

9.7.2. Flexural Strength

Six self-adhesive resin cements were selected: experimental automixed paste/paste self-adhesive cement, SmartCem™2 (Dentsply/Caulk), Rely X™ Unicem Aplicap (3M ESPE), MaxCem™ (Kerr), G-Cem (GC), BisCem™ (Bisco) and MonoCem™ (SHOFU). The rectangular mold (25 mm × 2mm × 2mm) was conditioned with a silicone lubricant, placed on a Mylar covered glass plate and slightly overfilled with the mixed cement. The second glass

plate was positioned on top of a piece of Mylar covering the mold. The two plates and mold were secured together using a binder clip and the cement was allowed to self-cure for 1 hr at 37 °C or light-cured in a Triad 200 for 2 minutes on each side. The specimens were removed from the molds, flash trimmed, and stored in deionized water at 37 °C for 23 hr. Flexural strength was determined in an Instron 4400R at crosshead speed of 0.75 mm/min.

Results:

24 hr Flexural Strength of SmartCem™2 vs. Competitive products

	Cure Mode*	SmartCEM2	Unicem	Maxcem	G-Cem	BisCem	MonoCem
Flexural Strength (MPa)	SC	67 ± 7	57 ± 12	49 ± 9	22 ± 7	25 ± 2	41 ± 5
	DC	107 ± 4	58 ± 4	69 ± 11	59 ± 6	35 ± 6	77 ± 16

*SC- Self Cure; DC- Dual Cure

9.7.3. Diametral Strength

Six self-adhesive resin cements were selected: experimental automixed paste/paste self-adhesive cement, SmartCem™2 (Dentsply/Caulk), Rely X™ Unicem Aplicap (3M ESPE), MaxCem™ (Kerr), G-Cem (GC), BisCem™ (Bisco) and MonoCem™ (SHOFU). The circular mold (6 mm in diameter and 3 mm in height) was conditioned with a silicone lubricant, placed on a Mylar covered glass plate and slightly overfilled with the mixed cement. The second glass plate was positioned on top of a piece of Mylar covering the mold. The two plates and mold were secured together using a binder clip and the cement was allowed to self-cure for 1 hr at 37 °C or light-cure in Triad 200 for 2 minutes on each side. The specimens were removed from the molds and stored in deionized water at 37 °C for 23 hr. Diametral tensile strength was determined in an Instron 4400R at crosshead speed of 10.0 mm/min.

Results:

24 hr Diametral Tensile Strength of SmartCem2 vs. Competitive products

	Cure Mode*	SmartCEM2	Unicem	Maxcem	G-Cem	BisCem	MonoCem
Diametral Tensile Strength (MPa)	SC	51 ± 4	32 ± 3	50 ± 3	17 ± 3	34 ± 1	48 ± 4
	DC	52 ± 4	38 ± 3	46 ± 9	42 ± 3	33 ± 3	49 ± 4

*SC- Self Cure; DC- Dual Cure

9.7.4. Radio-opacity

Radio-opacity of cured cements was measured in accordance with Caulk method FG-195-88 based on ISO 4049. A disc specimen with thickness of 1mm was prepared. The dental X-ray film of speed group D was positioned on a sheet of lead. The specimen and the aluminum step wedge were placed in the center of the film. The specimen, aluminum step wedge and film with X-rays at 65±5 kV at a target film distance of 400 mm, were exposed for a period of time that, after processing, the region of film beside the specimen and aluminum has an optical density of between 1.5 and 2. After developing and fixing the film, the optical density of the image of the specimen and that of each step of the aluminum wedge were measured using a densitometer. The resulting thickness of aluminum that was closest to that of the test chip was determined by taking readings on each side of the wedge that was the closest visual match. This value was reported as the radio-opacity.

Results:

The radio-opacity of five different shades SmartCem™2 was measured. Calibra® Esthetic Resin Cement was used as control. The radio-opacity of MaxCem™, Rely X™ Unicem and Fuji Plus (GC) were also tested for comparison. SmartCem™2 has radio-opacity of 2.0 for all five shades, exceeding ISO minimum of 1.0, 0.5 lower than for Rely X™ Unicem but 0.5 higher than MaxCem™.

Results: Radio-opacity of SmartCEM2 and Other Competitive products

Materials	Shades	Radio-opacity (mm Al)
SmartCEM2	Translucent	2.0
	Light	2.0
	Medium	2.0
	Dark	2.0
	Opaque	2.0
Calibra	Translucent	1.5
	Light	1.5
	Medium	1.5
	Dark	1.5
	Opaque	1.5
Maxcem	Clear	1.5
Unicem	Clear	2.5
GC Fuji Plus (powder/liquid)	Yellow	2.0

9.7.5. Film Thickness

The film thickness of the dental luting cements were measured in accordance with Dentsply Caulk Quality Method-071-90 as follows: A portion of material to be tested is placed between two glass plates of uniform thickness. A load of 15 kilograms is placed on the top plate. After 10 minutes, the plates are removed and measured. The difference in the thickness of the plates with and without the cement film is the film thickness of the cement.

The film thickness of self-adhesive resin cements (translucent shade) were tested. Results varied from 14.8 µm for Breeze to 29.2 µm for BisCem. SmartCEM2 seemed to have film thickness comparable to that of other major products.

Results:

Film Thickness of Self-Adhesive Cements

Product	Manufacturer	Delivery	Film Thickness (µm)
SmartCem2	Dentsply	Paste/paste Automix	19.6 ± 0.5
Maxcem	Kerr	Paste/paste Automix	20.0 ± 1
Rely-X Unicem	3M ESPE	Powder/liquid Capsule	23.2 ± 0.4
MonoCem	Shofu	Paste/paste Automix	17.6 ± 0.5
BisCem	Bisco	Paste/paste Automix	29.2 ± 0.8
Breeze	Pentron	Paste/paste Automix	14.8± 0.4
Multilink Sprint	Ivoclar- Vivadent	Paste/paste Automix	23.0 ± 1

9.7.6. Water Solubility and Water Sorption

Water sorption and water solubility of SmartCEM2 (SAC) with five different shades were tested in accordance with ISO and Dentsply SOP FG-270-92 in comparison with Maxcem and Unicem. Water sorption and water solubility of SmartCem™2 met the ISO limits. SmartCem™2 exhibited significantly lower water sorption than both Maxcem and Unicem and significantly lower water solubility than Maxcem.

Results:

Water Sorption and Solubility of Caulk SmartCem™2 vs. Maxcem and Unicem

	SmartCem™2					Maxcem	Unicem
	Translucent	Light	Medium	Dark	Opaque		
Water Sorption (µg/mm ³)	20.11 (1.39)	20.36 (0.39)	21.22 (0.31)	20.22 (0.54)	19.94 (0.76)	59.76 (5.56)	26.70 (0.78)
Water Solubility (µg/mm ³)	2.78 (0.82)	1.81 (0.65)	3.69 (1.32)	2.54 (0.55)	2.53 (0.64)	18.21 (2.99)	2.20 (0.30)

9.7.7. Water Expansion

The self-adhesive base/catalyst paste mixtures were placed in a Teflon mold and allowed to self-cure in 37 °C oven for one hour. The edges of the cured discs were lightly sanded to trim any flash. Two cross lines at 90° through the center of the chip were inscribed. The initial length of the lines was measured using a micrometer. The chips were stored in 0.9% sodium chloride aqueous solution at 37°C. The lines were measured again at various time intervals and the linear expansion (LE) was calculated according to the following formula:

$$\text{Linear Expansion} = (\text{Length after storage} / \text{Length before storage} - 1) \times 100$$

Results:

6-month Water-induced Linear Expansion of SmartCem2 vs. Competitive products

	SmartCEM2	Unicem Aplicap	Maxcem	BisCem	MonoCem
LE (% , mean ± s.d.)	0.35 ± 0.11	0.59 ± 0.03	0.75 ± 0.12	1.82±0.27	1.42 ±0.29

9.7.8. Working Time and Setting Time

The self-adhesive base/catalyst paste mixtures were mixed together and probed with a dental instrument. Work time at RT (23 °C) is recorded as the elapsed time from the start of mix to the point of paste forming a peak when the instrument is drawn through the paste. Set time, recorded at 23 °C or 37 °C, was the time elapsed from start of mix to the point of the material snapped sharply when sliced with Bard-Parker knife.

Results:

Work Time/Set Time of SmartCem2 and Unicem Aplicap and Maxcem

	SmartCem2	Unicem Aplicap	Maxcem
Work Time @23 °C	2'45"	2'15"	3'30"
Set Time @23 °C	5'00"	22'00"	5'00"
Set Time @37 °C	2'00"	4'15"	2'00"

9.7.9. Compatibility with LED and Halogen Curing Lights

Depth of cure for SmartCem™2 (SAC) when cured with both Spectrum® 800 (halogen light) and SmartLite® iQ™2 (LED light) was measured according to Dentsply method FG-751-80. A round Teflon mold (6 mm in diameter, 7 mm in height) was placed onto a strip of the transparent film on a glass microscope slide. The cement was dispensed into the mold, taking care to exclude air bubbles. The mold was slightly overfilled and a second strip of the transparent film was placed on top followed by a second microscope slide. The mold and strips of film were pressed between the glass slides to displace excess material. The mold was placed onto filter paper and the microscope slide covering the upper strip of film was removed. The light probe was placed against the strip of film and the material was irradiated for 10 seconds with either the Spectrum 800 at 550 mw/cm² or SmartLite iQ2. Immediately after completion of irradiation, the specimen was removed from the mold and the soft material was removed from the underside of the cured specimen with a razor knife. The hardness of both the topside and underside was measured using a Barcol Hardness Meter (medium).

If hardness of the underside was lower than 80% of that for topside, the underside of the specimen was sanded with 400 grit sandpaper until the required hardness was achieved. The thickness of the specimens was measured with a micrometer and the average value of five specimens was recorded as depth of cure.

Results:

Depth of Cure (mm) of SmartCem2

Shade	Halogen	LED
Translucent	4.2	3.4
Light	2.8	2.2
Medium	3.0	2.2
Dark	2.1	1.8
Opaque	1.3	1.3

9.7.10. Color Stability

Water Storage: A circular mold (20 mm in diameter and 1 mm in height) was placed on a Mylar covered glass plate and slightly overfilled with the mixed cement. The second glass plate was positioned on top of a piece of Mylar covering the mold. The two plates and mold were secured together using a binder clip and the cement was allowed to light-cure in Triad 200 for 2 minutes on each side. The specimens (n=5) were removed from the molds and stored in deionized water at 37 °C for 24 hr. The initial color values were measured in the CIE L*a*b* scale on a Greta Macbeth Color-EYE 3100. The color values were again measured after storage in 37 °C water for 3 days and 7 days. The total change in color, ΔE, were calculated.

Results:

Color Change of SmartCem2 Shades after Storage in Water

	Translucent	Light	Medium	Dark	Opaque
ΔE, 3 day storage	0.4	0.52	0.32	0.17	0.09
ΔE, 7 day storage	0.32	0.22	0.22	0.11	0.22

Ultraviolet Irradiation: A circular mold (20 mm in diameter and 0.5 mm in height) was placed on a Mylar covered glass plate and slightly overfilled with the mixed cement. The second glass plate was positioned on top of a piece of Mylar covering the mold. The two plates and mold were secured together using a binder clip and the cement was allowed to light-cure with Spectrum 800 @550 mw/cm² for 10" on each section, starting in the middle and then overlapping clockwise until the whole chip area had been cured. Specimen 1: After removal from the mold, one specimen was stored in the dark, dry in the oven at 37⁰C for 7 days; this is the reference specimen. Specimen 2: after removal from the mold, one specimen was stored in the dark, dry in the oven at 37⁰C for 24h. After this time, the specimen was removed from the oven and half of it was blanked off with aluminum foil. The specimen 2 was placed in Heraeus Suntest Unit, immersed in water at 37⁰C and exposed to UV radiation for 24 h. After exposure, the metal foil was removed, the specimen was transferred back to the oven at 37⁰C and stored in the dark, dry for 5 more days. The color of both halves of specimen 2 was compared with each other and with reference specimen 1. The color comparison was carried out in accordance with ISO 7491.

Results:

No noticeable color change was observed with all 5 shades of SmartCem2 among the specimens stored at the specified conditions.

9.7.11. Fracture Toughness

Fracture toughness of cured cements was measured in accordance with Caulk Method FG-339-00. The Teflon triangular prism molds with dimension 6 X6 X 6 X12 mm were slightly overfilled with cement paste. Any excess material was expressed from the mold by covering the material with Mylar sheet and glass slide with pressure. After the cement was cured, the specimen was removed from the mold and the flash was removed from top edges of the specimen first by cutting with a razor knife and then by carefully sanding using 600 grit sandpaper. A deep crack initiation point, approximately 0.1 mm deep, was made midway along the bottom edge of specimen using a razor knife. The specimens (n=6) were stored in

DI water at 37 °C for 24 hr and then placed into the mounting assembly with the crack initiation point aligned with the split line of the holder and secured in place by the two screw-tightened lids. The assembly was loaded in tension at a cross-head speed of 0.1 mm/min.

Results:

Fracture Toughness of SmartCem2 vs. Competitive products

Materials	Curing Mode	K_{IC} (MPa. m^{1/2})
SmartCem2	SC	0.99 (0.33)
	DC	1.08 (0.36)
Maxcem	SC	0.95 (0.22)
	DC	1.35 (0.45)
Unicem	SC	1.03 (0.26)
	DC	0.71 (0.20)
GC Fuji Plus	SC	0.45 (0.10)

9.7.12. Marginal Integrity

Principal Investigator: Roland Frankenberger

Report Author: Huaibing Liu

Testing Protocol:

Specimen Selection, Involved Materials and Tooth Preparation

Standardized Class II cavity preparation (MOD, 4 mm in width bucco-lingually at the isthmus, 3 mm in depth occlusally, 2 mm in depth at the bottom of the proximal box) were performed on human teeth. Proximal margins were located 1-2 mm above CEJ mesially, and 1-2mm below the CEJ at distal aspects.

The cavities were cleaned with pumice slurry and treated with SmartCem™2, Rely X™ Unicem Aplicap, MaxCem™ and Multilink Sprint (Ivoclar), respectively. Internal surfaces of the ceramic inlays were pre-treated with 5% hydrofluoric acid for 45s, rinsed with air-water spray for 60s, cleaned in an ultrasonic bath, dried, and then silanated with Monobond S for 5 min. Luting cements were polymerized with a Translux CL light-curing unit @600mW/cm². After finishing and polishing, the restored teeth were stored in distilled water at 37⁰C for 21 days. Impressions of the teeth were taken and a first set of epoxy resin replicas was made for SEM evaluation.

Functional Loading in a Chewing Simulator

Thermo-mechanical loading of specimens was then performed in an artificial oral environment. One specimen was arranged in one simulator chamber (Fig. 1) and obliquely occluded against a steatite antagonist for 100,000 cycles at 50N at a frequency of 0.5 Hz. The specimens were simultaneously subjected to 2500 thermal cycles between 5 and 55 ⁰C by filling the chambers with water in each temperature for 30s.

Analysis of Marginal Integrity

After the completion of 100,000 mechanical loading and 2500 thermal cycles, impressions of the teeth were retaken and another set of replica was made for each restoration. The replicas were mounted on aluminum stubs, sputter-coated with gold and examined under a SEM. The percentage “continuous margin” in relation to the individual observable margin was calculated as marginal integrity. Non-parametric statistical analysis was performed for pairwise comparisons at the 95% significance level.

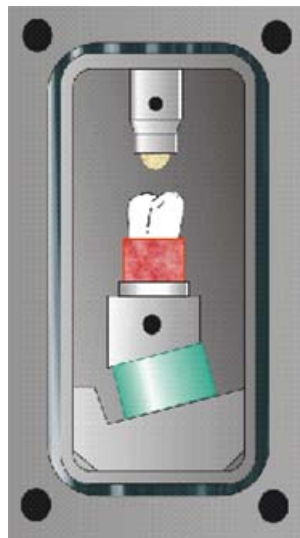


Fig.1. Arrangement of specimens in a chamber of the chewing simulator.

Results:

The results of dentin and enamel marginal integrity before and after thermo-mechanical loading (TML) are displayed in Tables 1 and 2. Same superscript letters within columns indicate no significant differences among groups ($p>0.05$).

Table 1. Enamel Marginal Integrity of Ceramic Inlays Luted with SmartCem2 vs. Competitive products

Luting Cement	Gap-free margins in enamel	
	before TML	after TML
	[%](SD)	[%](SD)
SmartCem2	95.5 (5.8) ^A	64.6 (9.6) ^A
Rely-X Unicem	91.4 (7.7) ^B	69.8 (15.0) ^A
Maxcem	96.9 (3.8) ^A	54.4 (11.6) ^B
Multilink Sprint	95.6 (4.8) ^A	56.6 (13.5) ^B

Table 2. Dentin Marginal Integrity of Ceramic Inlays Luted with SmartCem2 vs. Competitive products

Luting Cement	Gap-free margins in dentin	
	before TML	after TML
	[%](SD)	[%](SD)
SmartCem2	98.5 (1.5) ^A	83.1 (5.2) ^A
Rely-X Unicem	100 ^A	87.4 (5.7) ^A
Maxcem	96.4 (4.4) ^A	62.0 (13.0) ^B
Multilink Sprint	99.4 (1.2) ^A	67.6 (11.3) ^B

Conclusion: After thermo-mechanical loading, SmartCem™2 exhibited marginal integrity similar to Rely X™ Unicem and significantly better than MaxCem™ and Multilink Sprint.

10. References

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