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| 1. USU Principal Author (Last, First, Middle Initial) | Dekow, Matthew, W |
| 2. Academic Title | Comprehensive Dentistry Resident |
| 3. School/Department/Center | Uniformed Services University / Fort Hood AEGD-2 |
| 4. Phone 508-523-8773 | 5. Email matthew.w.dekow.mil@mail.mil |
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| Signature | [redacted] |

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**CPT Matthew Dekow, DMD**
**Fort Hood AEGD-2**
Uniformed Services University
Date: **05/09/2017**

Effects of silanation of ceramic crowns on bond strength using a new bioactive cement

A Thesis

Presented to the Faculty of the Advanced Education in General Dentistry, Two-Year Program,

United States Army Dental Activity, Fort Hood, Texas

And the Uniformed Services University of the Health Sciences – Post Graduate Dental College

In Partial Fulfillment of the Requirements for the Degree of

Master of Science in Oral Biology

By

Matthew Dekow, CPT USA DC

May 2017

Effects of silanation of ceramic crowns on bond strength using a new bioactive cement

A REPORT ON

Research project investigating the effect of silanation on the bond strength of a new bioactive cement (Ceramir C&B) to lithium disilicate blocks and how it compares to the bond strength of RelyX Unicem to lithium disilicate blocks

By

Matthew Dekow, CPT, DC, USA

D.M.D., Boston University- Boston- 2014

Staffed By

Michael Mansell, LTC, DC, USA

Fort Hood, Texas

May 2017

## ABSTRACT

**Purpose:** The purpose of this study is to 1) examine and compare the bond strength of silanated Emax crowns and non-silanated Emax crowns cemented with Ceramir calcium aluminate cement, 2) examine and compare the bond strengths of Ceramir cement and RelyX Unicem cement to Emax crowns 7 days post cementation, and 3) determine whether silanation makes a difference overall in bond strengths of bioactive cements.

**Methods:** Four groups total, each with 19 samples were created for a total of 76 samples. Two of the groups were cemented with Ceramir and the other two groups were cemented with RelyX Unicem. Within these 2 groups, half were silanated and the other half were not silanated. All samples were created from Emax blocks of different shades, machine polished, and etched with 9.6% hydrofluoric acid. Uniformity of sample surfaces was confirmed under a microscope. Cement buttons were placed on each sample surface and samples were placed in a buffering solution for 7 days. Samples were then tested using the shear bond strength test and maximum bonds strengths were recorded for each sample in megapascals. Modes of failure were determined using a microscope and failures were placed into one of three categories: 1) adhesive, 2) cohesive, or 3) mixed.

**Results:** A significant difference in bond strengths ($\alpha$=0.05) was found between the RelyX group and the Ceramir group and the silanated group and non-silanated group. Within the Ceramir group, there was no difference between the silanated group and the non-silanated group. Within the RelyX group, there was a difference between the silanated group and the non-silanated group.

**Conclusions:** The bond between RelyX Unicem and Emax was significantly greater than the bond between Ceramir and Emax. This bond using RelyX was increased when the sample was silanated before cementation. Within the Ceramir groups, there was no change in bond strength after the samples were silanated.

ACKNOWLEDGMENTS

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- Dr. Wen Lien of the U.S. Air Force for assisting with the protocol submission, data collection, and statistical analysis

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INTRODUCTION

Ceramir C&B is a new bioactive luting cement that was released to the market recently. The manufacturer claims that it is a luting cement that also forms a bond with the tooth and the crown. Indications for use include PFM, lithium disilicate, zirconia, and gold inlays/onlays. They claim that this is the closest cement that mimics normal tooth structure, and has the potential to regenerate tooth structure. It's biocompatible, has exceptional retentive strength, and is easy to use and handle.[12] Also of importance is its ability to seal marginal gaps.[2] The instructions for use are rather simple and no silanating agent is needed when cementing lithium disilicate crowns, which makes the cementation process even easier and cheaper for the clinician. The purpose of this study is to test the bond strength of the cement to lithium disilicate crowns that have been silanated compared to those that have not been silanated.

The setting reaction of Ceramir cement is rather unique. The pH is acidic when first placed (pH~4), after one hour becomes neutral, and after 4 hours it becomes basic (pH~8.5). This is all due to the acid base reaction of the glass ionomer component.[12] Glass ionomers are known for leaking over time, but the calcium aluminate in Ceramir fixes the glass ionomer structure, preventing the cement from leaking.[2] This property along with the basic pH of the cement allows apatite to form toward the tooth surface.[13] This allows the cement to seal or reseal marginal gaps.[2]

In military dentistry, it is important to use materials that will maximize soldier readiness and decrease lost time to multiple appointments. Materials that are biocompatible, cause minimal to no sensitivity, and are easy and fast for the clinician to use are desirable. Ceramir C&B cement would be an ideal cement for the military to use due to the time constraints we face. Eliminating the silanation step in the cementation protocol allows for the elimination of a step that could introduce error in the cementation technique. This would lead to less crown failures for soldiers, a decreased loss of manpower hours, and a higher soldier readiness for the Army. Also, Ceramir cement is about 50% cheaper than conventional resin cements (i.e. RelyX Unicem), which would decrease the burden on unit budgets.

There are many types of cement on the market today. Depending on the clinical application, the clinician has to decide which cement would work best. The two main classes of cements that exist today are luting cements and resin adhesive cements. Ceramir C&B is a new class of cement that is bioactive. This means that it has luting properties but also forms a bond with both the tooth and the crown. It is a nanostructurally integrated bioceramic, which means the formation of hydroxyapatite minimizes the leakage between tooth and material over time. This cement is composed of two stable hydrates which are gibbsite and katoite. The gibbsite transforms over time into crystalline gibbsite which is responsible for the nanointegration with the tooth structure. This bond is on the nano-level because it is a mechanism that is built on surface energy and mechanical interlocking. Ceramir is made up of both calcium aluminate and glass ionomer. Research exists on the biocompatibility, microleakage, and bond strength to tooth of biocompatible cements, but not much research exists on the bond strength to the crown. There are limited studies on how silanation affects the bond strength of the Ceramir cement to the crown and how the bonding of the cement to the crowns compares to other cements.

Bioactive cements can seal or reseal artificial marginal gaps in simulated aqueous physiological conditions by fostering build-up of nano-crystals that integrate with the tooth.[2] They can significantly improve marginal stability of these crowns, which is important for our patients who are deployed. Marginal leakage and recurrent caries is a problem for our deployed soldiers. The true definition of a bioactive material is one that forms a surface layer of an apetite-like material in the presence of an inorganic phosphate solution.[18] One example of this is fluoride releasing dental materials.[18] Glass ionomers, which have been around for a while, do not truly fit this definition because they do not form hydroxyapatite.[18]

Even though resin-modified glass ionomer does not fit the definition of a bioactive material, calcium hydroxide and mineral trioxide aggregate are two bioactive materials that have been around for a long time. The exact mechanism of Ceramir cement is proprietary and unknown to the clinician. According to Kugel, the mechanism is similar to calcium hydroxide, which dissociates into calcium and hydroxyl ions.[18] These calcium ions reduce capillary permeability and lessen the serum flow. It causes mineralization by decreasing the levels of inhibitory pyrophosphates.[18] We do not know if Ceramir works in this fashion, but we do know that the cement has calcium aluminate, so calcium ions may be involved with the bioactivity of the material.

Silane, or silane coupling agents, are synthetic hybrid inorganic-organic compounds which act as bifunctional molecules. They are used to promote adhesion between dissimilar materials. The following shows the structure of silane:

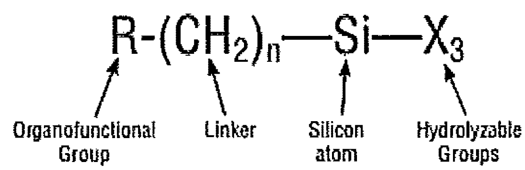



**Figure 1.** Silane structural makeup

The X is the "reactive" group that bonds with inorganic materials, which in dentistry would be the glass content in crowns. The R is the "reactive" group that bonds with organic materials, such as the resin in the cement.

Silanation is an important step for bonding to the lithium disilicate material itself. Ceramir claims that the silanation step is not needed when cementing Emax with Ceramir cement. However, the Emax Instructions for Use say the silanation step is needed for cementation when using a resin or bonding cement. Studies not only show that silanated crowns have better bonding, they also had higher fracture resistance.[8] According to Ceramir, eliminating the silanation step does not seem to affect the properties of Ceramir cement. The retentive properties are still excellent, and no retentive failures have been reported.[11]

The protocol set forth to test the shear bond strength of the different groups in this study was created using the ISO standard 29022.[19] Even though extracted teeth were not used in this study, ISO 29022 reviews how to prepare the restorative samples and how to store them. It also sets the standard for shear bond testing the samples.

PURPOSE

The purpose of this study is to 1) examine and compare the bond strength of silanated Emax crowns and non-silanated Emax crowns cemented with Ceramir calcium aluminate cement, 2) examine and compare the bond strengths of Ceramir cement and RelyX Unicem cement to Emax crowns 7 days post cementation, and 3) determine whether silanation makes a difference overall in bond strengths of these materials.

## HYPOTHESIS

Research questions: Will there be a significant change in bond strength between Ceramir and RelyX Unicem cement? Will there be a significant change in bond strength in the samples that were silanated versus the ones that were not silanated? Within each cement group, will silanation affect the bond strength to lithium disilicate?

Null hypothesis #1: There will be no difference in bond strength between the Ceramir and RelyX Unicem cements.

Null hypothesis #2: There will be no difference in bond strength between the silanated and non-silanated groups.

Null hypothesis #3: There will be no difference in bond strength between the silanated and non-silanated samples within each cement group.

## MATERIALS AND METHODS

ISO Standard 29022 was used to create the protocol of this study.[19] Nineteen blocks of IPS e.max CAD blocks were obtained from the manufacturer from four different lots. The samples were made up of various shades (Table 1). While in the meta-silica state, each block was sectioned into four samples (each 3.5mm thick) using the Buehler Isomet 5000 linear precision saw (SN 15651060, Picture 2). All blocks were within their expiration dates. The blade speed for the sectioning was set to 700rpm with a feed rate of 5.0mm/min. The blade thickness was 0.508mm. Copious amounts of water was used when sectioning the blocks (Picture 3).

Each sample was then fired using the Vita Vacumat 6000 M oven (SN 1320101198, Picture 4). Eight samples were placed on a plate and mounted with IPS Object Fix Auxiliary firing paste (Lot V10077, Picture 5). They were fired using the E.max CAD/CAM crystal glaze setting on the oven, which holds it at 840 degrees Celsius for 7 minutes (Picture 6).

Once crystalized, the samples were fixed in ring formers using EpoxiCure 2 Epoxy Resin (Item #20-3430-128) and EpoxiCure 2 Epoxy Hardener (Item #20-3432-032). Two samples per ring were placed on a glass slab coated with Buehler Release Agent (Item #20-8185-016, Picture 7). The resin was allowed to set for 24 hours. Once set, the back of each sample was marked using a permanent marker.

The samples were then polished (6 rings at a time) using the Buehler Automet 3 Powerhead and Ecomet 6 Variable Speed Polisher (SN 586-A3P-00411). CarbiMet SiC Abrasive paper was used starting with a 320 grit (30-080320), then 400 grit (30-08-0400), and finishing with a 600 grit (30-08-0600, Picture 8). On each grit, the samples were run through 2 cycles at 220rpm using 10 pounds of pressure. The direction of the motor would change direction for each cycle. Each sample was then evaluated under a microscope to verify that all the resin was removed from the ceramic surface (Pictures 9 and 10). Samples that had resin remaining were run through another polishing cycle using all grits.

Samples were then separated into the four following groups:

1) Emax cemented with Ceramir cement (no silanation)
2) Emax cemented with Ceramir cement (silanated)
3) Emax cemented with RelyX Unicem cement (no silanation)
4) Emax cemented with RelyX Unicem cement (silanated)

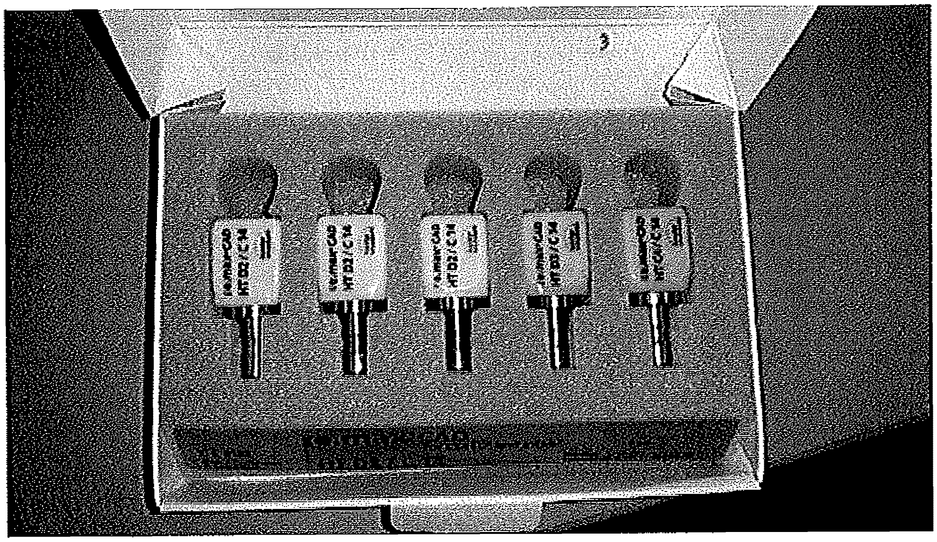
Every sample was etched for 20 seconds using Pulpdent Porcelain Etch Gel 9.6% Hydrofluoric Acid (Lot 160226). The acid etch was then rinsed off and the sample was steam cleaned. The samples were air dried. Pulpdent Silane Bond Enhancer (Lot 160128) was brushed onto the samples in the silanation group and left to react for 60 seconds. Any remaining silane was gently blown off with moisture and oil free air.

Cement buttons (RelyX Lot 615353, Ceramir Lot 101395) of each cement were then placed on the samples using an Ultradent Jig (Picture 11, 12). Cement was cured for 24 hours before placing samples in Phosphate Buffered Saline (Lot SLBP1037V). This Sigma solution contains 0.01M phosphate buffer, 0.0027M potassium chloride, and 0.137M sodium chloride with a pH of 7.4. Samples remained in this solution for 7 days at room temperature.

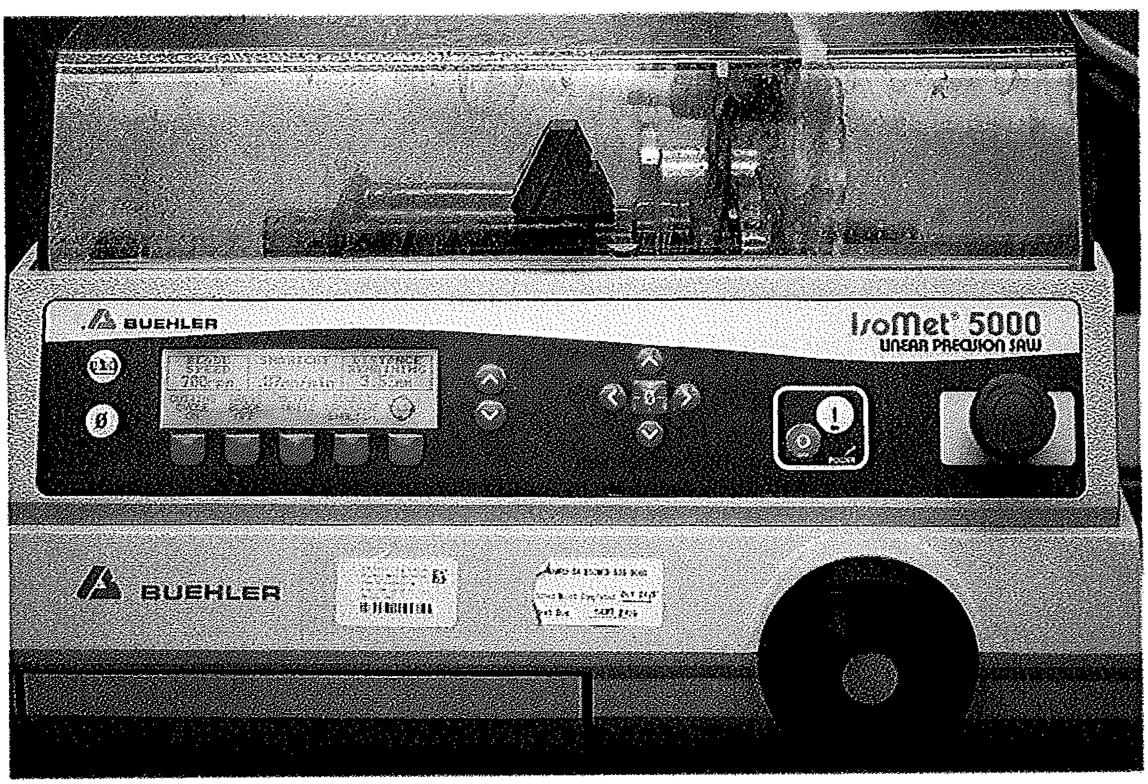
After 7 days, the samples were shear tested on a MTS Alliance RT/5 (SN M10162408, Picture 13). The crosshead speed was set to 0.01mm/sec (Picture 14). The test for each sample was terminated when the machine detected a break. The value of the max force (N) needed to de-bond the sample was recorded. The area of the broken cement button was then measured using calipers and this value was recorded in mm. The stress values were then calculated by dividing the max force by the area of the broken button. This value was recorded in MPa.

Each sample was analyzed under a light microscope (SN 9L13825) to determine the mode of failure (adhesive, cohesive, or mixed, Picture 15). If little to zero cement remained on the sample, it was classified as an adhesive failure (Picture 16). In an adhesive failure, there is a failure of the bond between the cement and the sample, but the cement and sample remain intact. If the entire surface was still covered in cement, it was a cohesive failure (Picture 17). In a cohesive failure, there is a failure within the structure of the cement, and some of the cement remains bonded to the sample. And if there was a moderate amount of cement remaining with voids of no cement, it was a mixed failure (Picture 18).

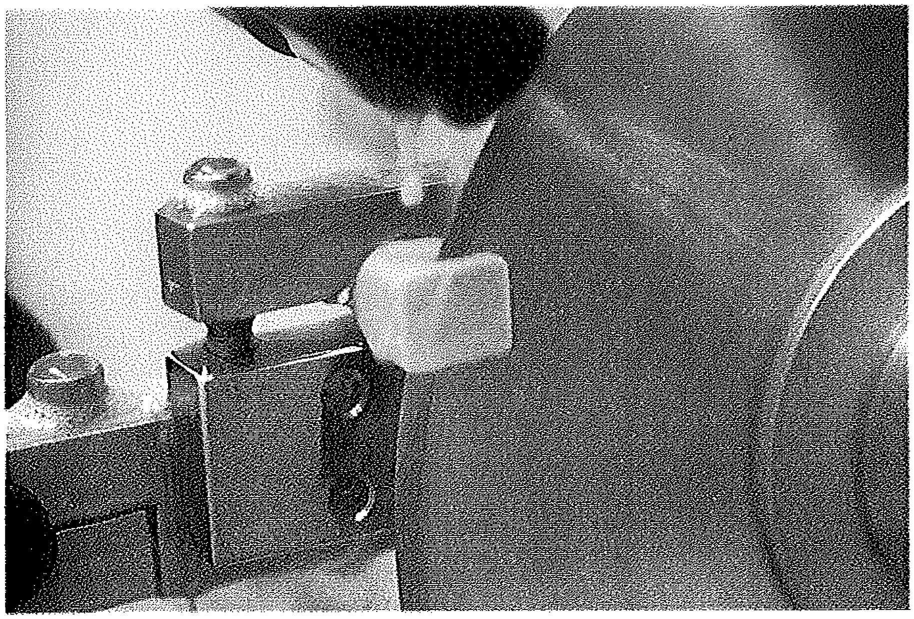
TABLE AND FIGURES



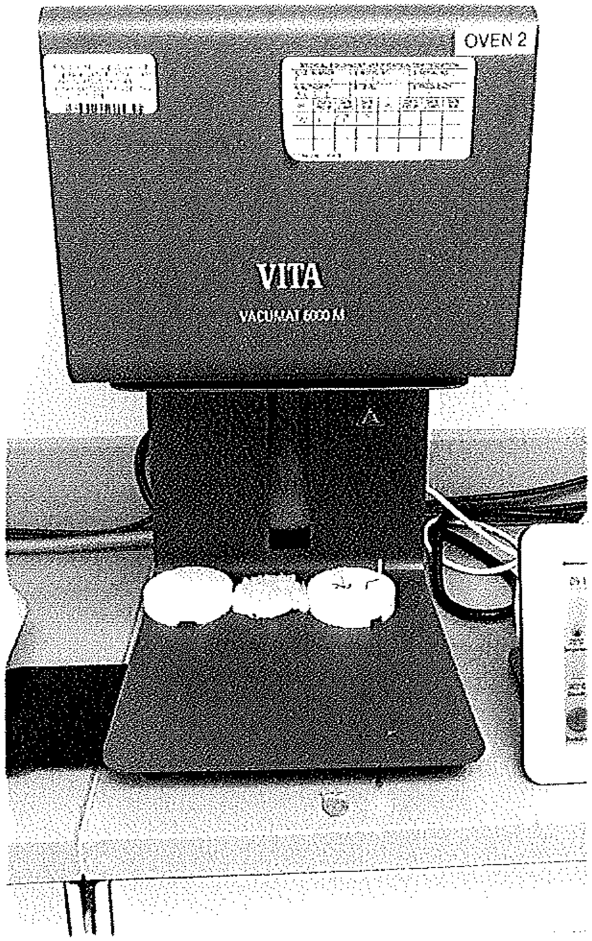
**Picture 1.** Emax sample blocks



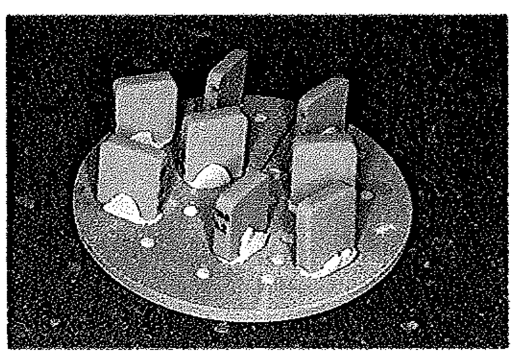

**Picture 2.** Buehler Isomet 5000 Linear Precision Saw



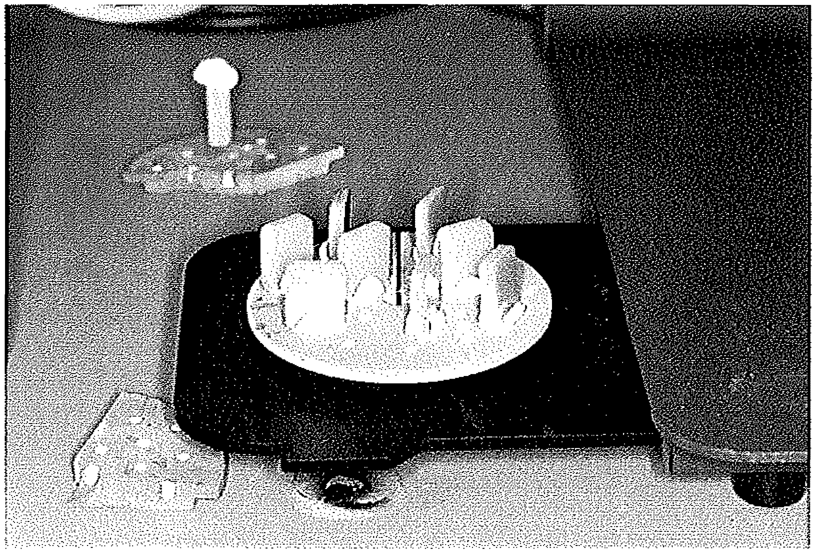
**Picture 3.** Close up of saw creating slice



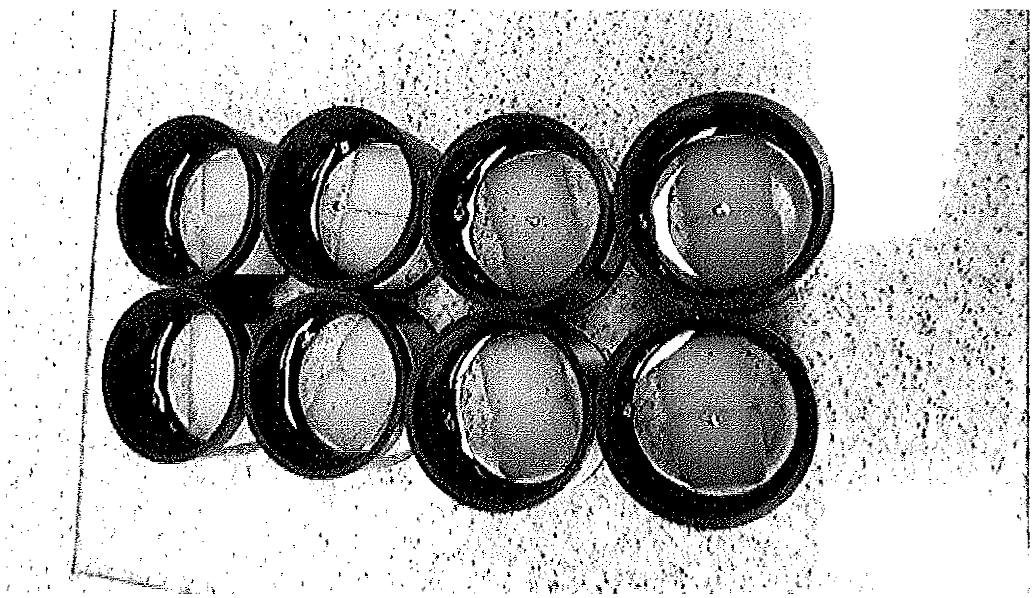

**Picture 4.** Vita Vacumat 6000 M Oven



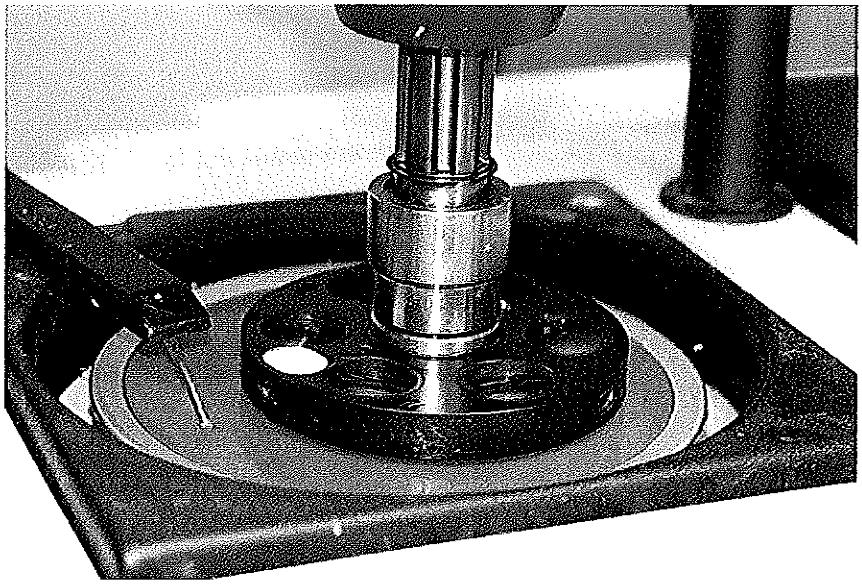
**Picture 5.** Sample preparation for firing



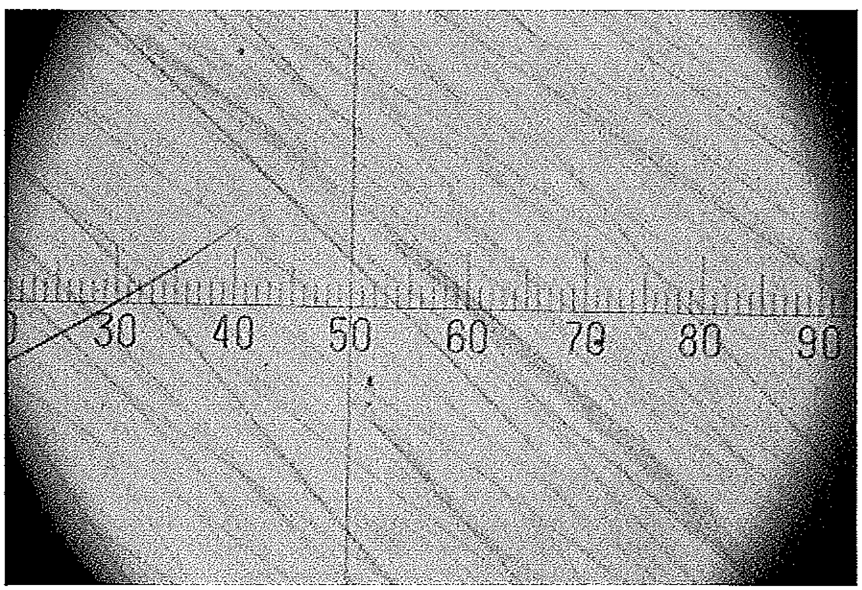
**Picture 6.** Samples after firing



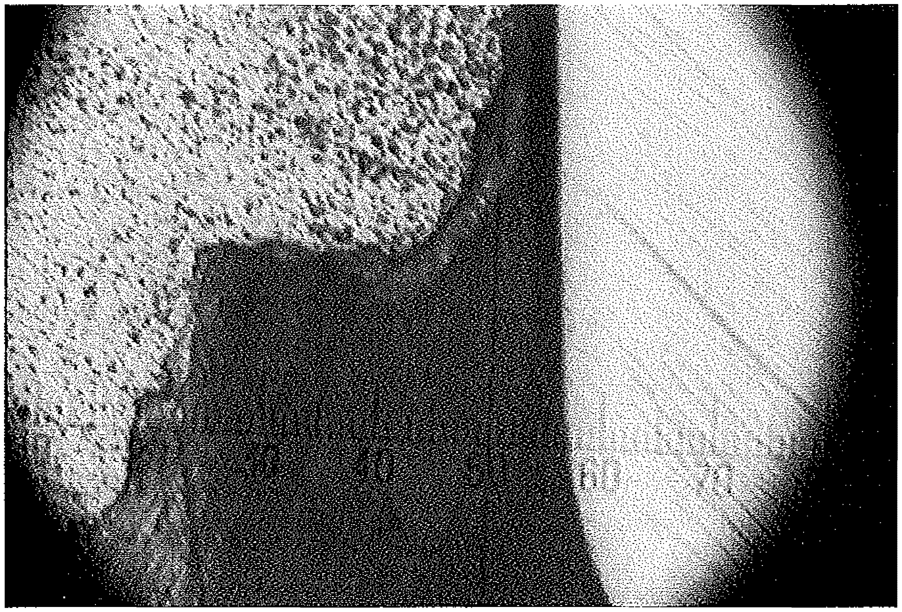
**Picture 7.** Samples setting in epoxy resin



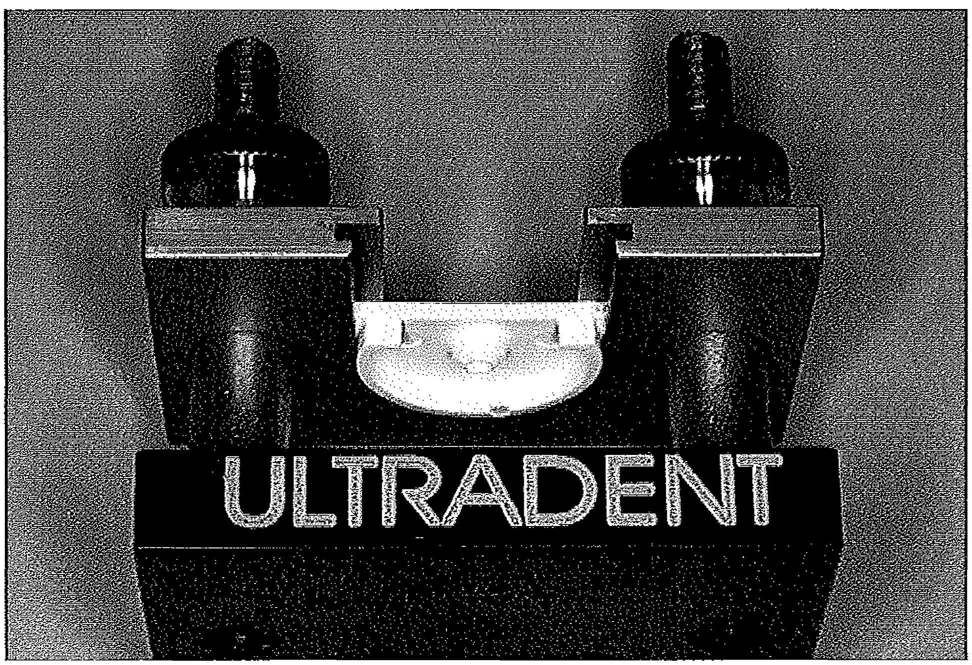
**Picture 8.** Samples polished with copious amounts of water



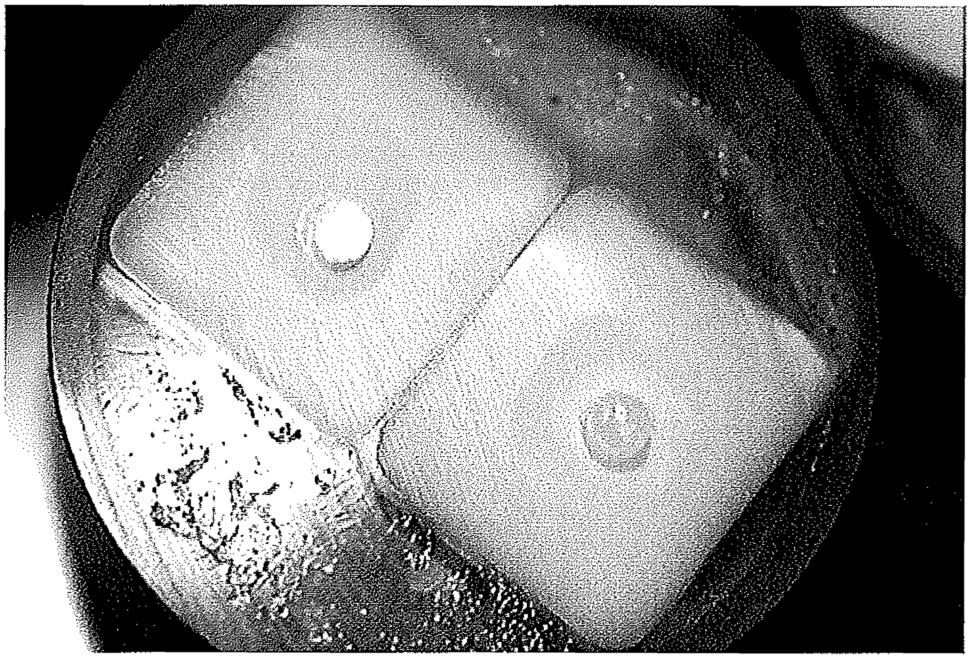

**Picture 9.** Sample with all resin removed



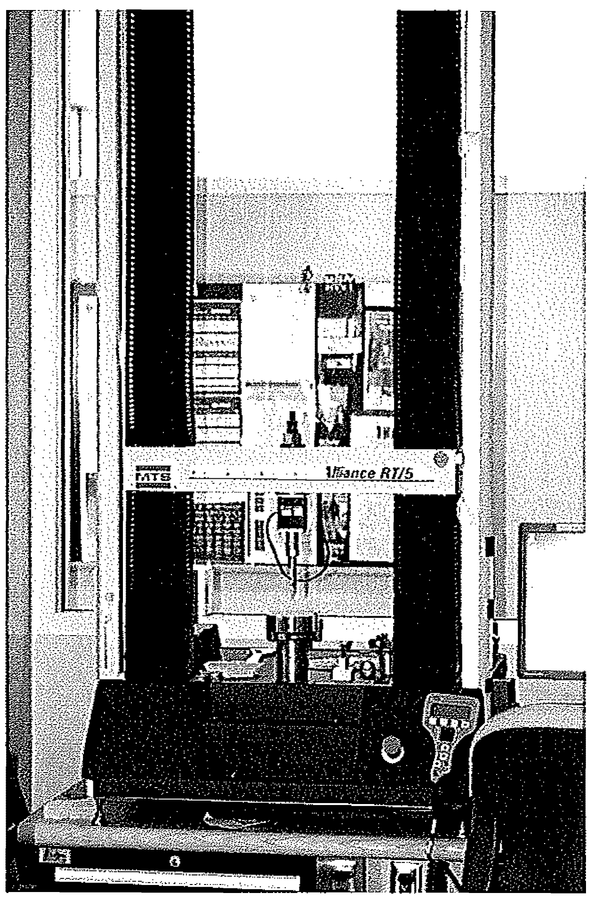
**Picture 10.** Sample with resin remaining



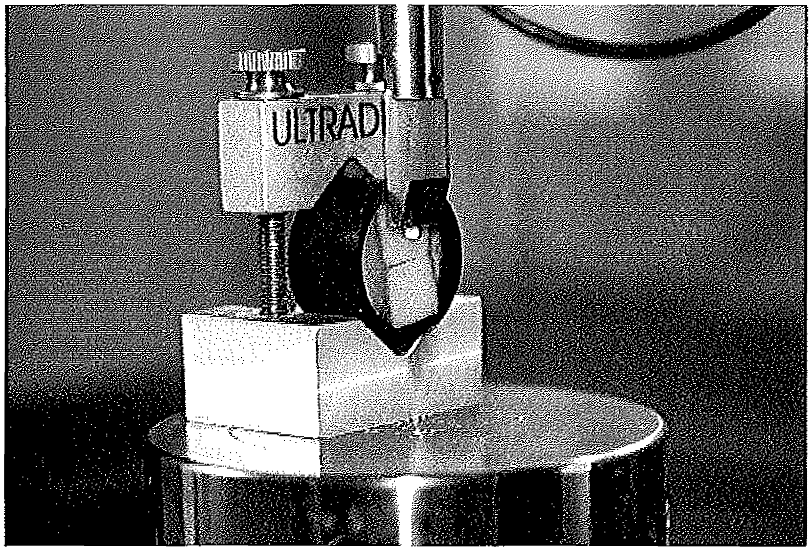

**Picture 11.** Jig used to create cement buttons



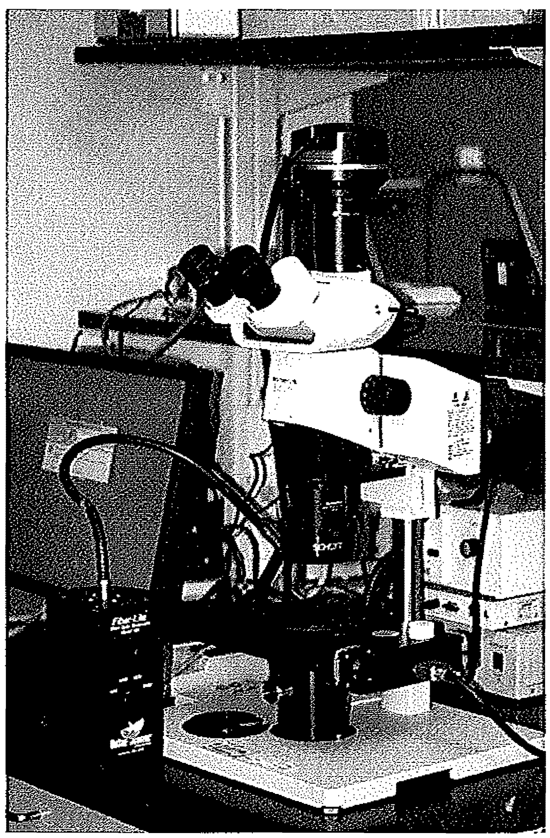
**Picture 12.** Cement buttons on E.max blocks (top left: Ceramir, bottom right: RelyX Unicem)



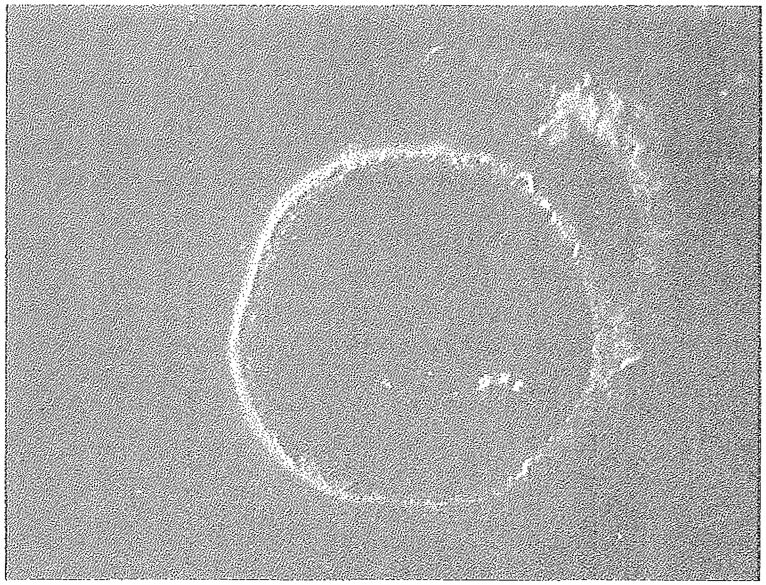

**Picture 13.** MTS Alliance RT/5 Shear Tester




**Picture 14.** Cross head on shear tester

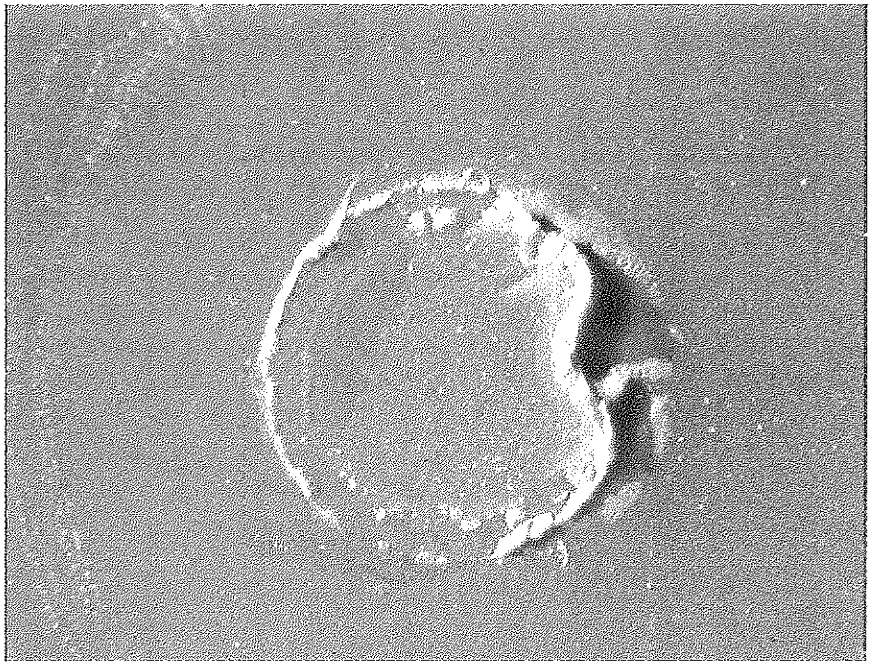


**Picture 15.** Microscope used to analyze failure modes



**Picture 16.** Adhesive failure

**Picture 17.** Cohesive failure



**Picture 18.** Mixed failure

## RESULTS

All 76 samples were shear tested and the force values in N were recorded. If the sample de-bonded before being shear tested, the value in N was recorded as zero. All of the zero recordings were in the Ceramir group. There were no zero values recorded for the RelyX group. Many of the cement buttons in the Ceramir group debonded shortly after cementing them on. A gentle stream of air or sudden movement would easily debond some of the Ceramir samples. Any dislodgement before testing was recorded as a zero.

All of the Force data was converted to stress values using the following formula from ISO29022 (19):

$$\sigma = F/A_b$$

where

$\sigma$ is stress, expressed in MPa;

$F$ is force, expressed in N;

$A_b$ is bonding area, expressed in $mm^2$.

The following table shows the average stress values that were calculated for each group in MPa.

| Silanated | Ceramir | RelyX |
|---|---|---|
| No | 0.027847 | 5.568823 |
| Yes | 0.026603 | 7.1867 |

**Table 1.** Average recorded stress values

The following chart is a graphical representation of the values in Table 1. Note that a secondary access is displayed because the Ceramir values were much lower than the RelyX values:

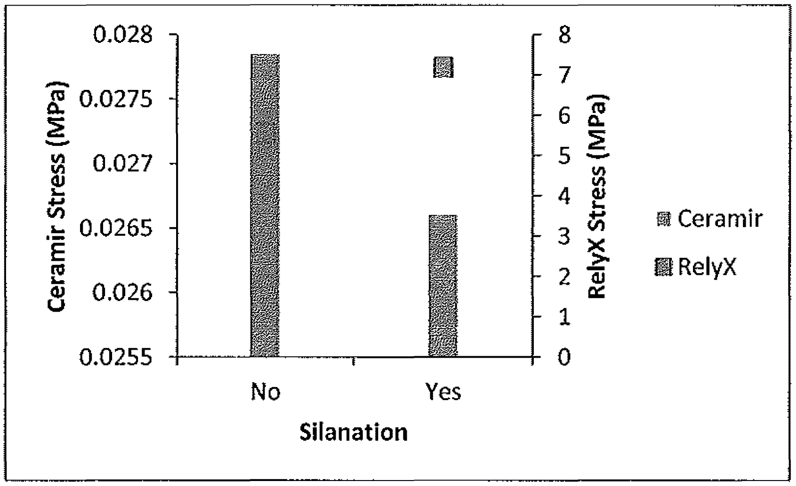



**Figure 1.** Average recorded stress values

A Tukey analysis was performed to determine statistical differences between the four groups. Three statistically different groups were identified using this analysis. All the Ceramir samples were grouped into the same group because no difference in silanation was detected:

**LSMeans Differences Tukey HSD**

α= 0.050 Q= 2.63006

| Level | | | | Least Sq Mean |
|---|---|---|---|---|
| Yes,RelyX | A | | | 7.1867004 |
| NO,RelyX | | B | | 5.5688229 |
| NO,Ceramir | | | C | 0.0278473 |
| Yes,Ceramir | | | C | 0.0266026 |

Levels not connected by same letter are significantly different.

**Table 2.** Tukey analysis

If just the silanation is analyzed without regard to the type of cement, there is a statistical difference between the samples that were silanated and those that were not:

(  (

**Silaned**

**LSMeans Differences Student's t**

| **Level** | | | **Least Sq Mean** |
|---|---|---|---|
| Yes | A | | 3.6066515 |
| NO | | B | 2.7983351 |

Levels not connected by same letter are significantly different.

**Table 3.** Least square mean differences between silanation groups

If just the type of cement is looked at without regard to silanation, there is a statistical difference between the Ceramir group and the RelyX group:

**LSMeans Differences Student's t**

α= 0.050 t= 1.99346

| **Level** | | | **Least Sq Mean** |
|---|---|---|---|
| RelyX | A | | 6.3777617 |
| Ceramir | | B | 0.0272250 |

Levels not connected by same letter are significantly different.

**Table 4.** Least square mean differences between cement groups

Most of the failures for the Ceramir groups were classified as adhesive failures (66%, Table 3). Many of the RelyX failures were mixed failures (76%, Table 3). Cohesive failures were rare.

| Failure Mode | Ceramir | RelyX |
|---|---|---|
| Adhesive | 66% | 24% |
| Mixed | 28% | 76% |
| Cohesive | 8% | 0% |

**Table 3.** Failure modes and their percentages

DISCUSSION

The results show that Ceramir is a far less superior cement than RelyX Unicem with regards to sheer bond strength. It is important to note that it may be an adequate luting cement, but Ceramir does not even compare to many of the bonding resin cements on the market. Table 1 and Figure 1 both show the significant difference in bond strengths between the two cements. The bond strength of the RelyX cement is 200 times that of the Ceramir cement.

During the study, many of the Ceramir buttons debonded shortly after creating them. Many others debonded when placed in the buffering solution. When this happened, the value for that sample number was recorded as a zero. Because Ceramir is a brittle cement, the design study for testing this cement may have been inadequate. Cements are only supposed to be used as a very small film thickness (maximum thickness equals 25um). Ceramir might be an adequate cement if used at this thickness because the brittleness will not matter. In addition, if the prep has good retention form, the brittleness of the cement will be less of a factor. One possible reason the RelyX Unicem cement did not debond so easily is because it is not very brittle when at a larger thickness. Keeping this in mind, a different design study could be used instead to get more accurate results for the Ceramir cement.

One possible design study would include using the cements at a smaller thickness. This may include sandwiching the cements between the ceramic block and a resin modified glass ionomer. The cement would bond these two materials together. Then the resin modified glass ionomer could be shear bond tested to failure. This may give higher shear bond strengths for the Ceramir cement because it would be used in a way that is more realistic.

Since Ceramir is a very brittle material when thick samples are created, the measurements for the surface area may be inaccurate. Calipers were used to measure the diameter of each failed cement button. Miniscule amounts of Ceramir cement would chip off when taking this measurement, which could lead to a smaller surface area than what was actually bonded to the lithium disilicate. This error could have artificially increased the stress values on the Ceramir data. However, this error is not significant because the force values on the Ceramir cement were so low that this small decrease in area would not make a significant difference in the stress values.

We know that silanation is an important step when bonding lithium disilicate crowns with resin cements. A study by Maruo shows that silane (8-methacryloxyoctyl trimethoxy silane) does increase the initial bond strength between lithium disilicate and cement.[14] It acts to increase the bond between the cement and the ceramic. Therefore, it was encouraging to see that it did make an overall difference in the bond strengths. Table 3 shows the statistical significance in bond strengths with the least square mean values. Table 1 clearly shows that RelyX Unicem had a significant increase in bond strength when the sample was silanated. It also shows that it may have hurt the bond strength of the Ceramir samples, although this is not statistically significant. Because silanation lowered the bond strength of the Ceramir cement, it may show that Ceramir may have no bond at all to the lithium disilicate.

There are many studies that try to explain the mechanism of action of silane on bonding surfaces. A study by Umer[15] tries to explain the effect of silane on dentin protease activity, which is significant because Ceramir claims to be a bioactive cement. In their study, they showed that a quaternary ammonium silane has antibacterial effects which resist dentin collagen degradation. They claim the silane not only improves bonding strength but also inhibits endogenous matrix metalloproteinases, which are responsible for breaking down the demineralized dentin. Using silane as a protease inhibitor will improve the durability of the cement bond and may increase hydroxyapatite formation.

One study by Lise shows that when silane was omitted from cementation protocols, the results were significantly lower than the samples that were silanated.[16] This study by Lise also showed that a short amount of storage time did not affect the bonding values. After three weeks of storage, there were no significant differences in shear bond strength, but after six months of storage, there were significant differences in the groups that were treated with silane compared to the groups that had no treatment. In the present study, the samples were tested one week after bonding. This eliminates the storage time variable from the results so that we are more easily able to compare the silanated groups with the non-silanated groups.

Composite restorative materials also benefit from silane treatment.[17] This mictrotensile bond strength study by Visuttiwattanakorn uses a two-way ANOVA test to analyze the results, much like what was done in the present study. The mean of all four groups was calculated and recorded in Table 1. As this study by Visuttiwattanakorn claims, using the two-way ANOVA test allows the researcher to evaluate the bond strengths and to compare the two variables individually.

More studies need to be completed before it can be determined if Ceramir would be a good cement for the military to use on its patient population. Debonding studies using actual crown preps would help determine the reliability and dependability of this cement (show gold crown Ceramir study here). Even though Ceramir is very cost effective, it would not be worth using if multiple debonds were occurring due to its lack of bond strength.

It is not surprising that most of the Ceramir failures were adhesive failures. With bond strength 200 times lower than RelyX Unicem, we would expect to see adhesive failures. This data is very subjective and is mainly determined by the amount of cement left on the sample. If most of the cement was off of the sample (>10% remaining on the sample) this was classified as an adhesive failure. If about 50% was remaining on the sample, this was classified as a mixed failure. And if greater than 80% was left on the sample, this was classified as a cohesive failure. There were not many classified as cohesive failures. Table 3 shows that Ceramir had 0% cohesive failures and this makes sense due to its brittleness. Given the difficulty of keeping the Ceramir buttons in-tact, we would expect to see 0% cohesive failures.

Bioactivity studies usually test the bond between the cement and the tooth structure, but in this study the bond between the cement and the ceramic was tested. A bioactive material is defined as one that forms a surface layer of an apetite-like material in the presence of an inorganic phosphate layer.[18] This is mostly considering the interaction between the tooth and the cement, and has little to do with the interaction between the cement and ceramic. Even though the bond to the ceramic was not that significant, it still may be a good cement to use after studies are performed on the bond to the tooth structure itself. Ceramir seals gaps which can be important when using milled emax crowns. There may be more space between the margin of the tooth and the crown margin, so increasing hydroxyapatite at this interface may help in reducing secondary caries.

Ceramir's bond to the tooth structure is rather unique in that it forms hydroxyapatite at 14 and 28 days after cementation.[18] The present study tested the samples after 7 days, possibly not allowing for HA production. Research shows that this HA formation on damaged tooth structure has benefits that include reducing the ability of secondary caries to form.[18] The clinician will have to weigh the risks and benefits of using a bioactive cement such as Ceramir. Even though the bond to the restoration may not be that great, the bioactive bond and HA formation at the tooth structure may have beneficial clinical implications for certain patient populations.[18]

Overall, more studies need to be completed using new bioactive cements including Ceramir. Until more studies show the clinical effectiveness of these cements, they should not be used in the military environment. They may be used as luting agents, but different studies with different designs need to be performed before this can be determined. Silanation does play a major role in bonding, but not with Ceramir cement due to its initial lack of bond strength.

CONCLUSION

The results of this in-vitro study lead one to conclude:

1. There is a statistically significant difference in the bond strengths between Ceramir and RelyX Unicem cements.
2. Silanation does have a positive effect on bond strength, independent of the cement being used.
3. Within the Ceramir group, there is no statistically significant difference in bond strength between the silanated and non-silanated samples.
4. Within the RelyX Unicem group, there is a statistically significant difference in bond strength between the silanated and non-silanated samples.

The findings in this study lead to the rejection of null hypotheses #1 and #2. Statistically significant sheer bond strength was noted in those groups. Null hypothesis #3 is accepted for the Ceramir groups but is rejected for the RelyX Unicem groups.

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