MICROTENSILE BOND STRENGTH OF NEW PASTE/PASTE RESIN-MODIFIED GLASS IONOMER CEMENT SYSTEMS: THE EFFECT OF DENTIN PRETREATMENT

by

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DEDICATION

This thesis is dedicated to my parents, my wife, my daughter, my brothers, and my sisters for their support, love, and patience which were my inspiration to successes.

ACKNOWLEDGMENTS

First, I want to thank the God for giving me the health to continue my study and reach this level of education.

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INTRODUCTION

There is an increased demand for aesthetic restorations in the dental practice. As a result, research has focused on the development of tooth-colored restorative materials such as resin-based composites, glass ionomers (GIC) and resin-modified glass ionomers (RMGIC).[1-3] The ultimate goal has been to develop restorative materials that can produce aesthetically pleasant restorations with adequate mechanical properties to withstand occlusal load forces.[3]

In order to improve the clinical performance of RMGIC, 3M ESPE and GC America have introduced paste/paste resin-modified glass ionomer cements, *Ketac™ Nano and* Fuji Filling™ LC, respectively. The Ketac Nano is claimed to possess better aesthetics while Fuji Filling LC claims to have better bonding properties. Both companies developed non-rinse substrate conditioners (i.e., Ketac Nano Primer-3M ESPE and GC Self-Conditioner-GC America) that should be used with these new paste/paste materials instead of the conventional polyacrylic acid. Both manufacturers have also recommended the use of this novel substrate conditioner with the previously marketed RMGICs. They claim it will enhance the bond of these materials.

The purpose of this study is to investigate whether the use of novel non-rinse conditioners as substrate pre-treatment and the new paste/paste RMGIC will affect the microtensile dentin bond strength of the material in comparison to the traditional resin-modified glass ionomer cement with polyacrylic acid as a surface substrate pre-treatment.

LITERATURE REVIEW

HISTORICAL BACKGROUND

Wilson and Kent were the first developers of conventional glass ionomer in 1969 at a government laboratory in London.[4] Conventional GIC is a cement material that results from an acid base reaction. Fluoroaluminosilicate glass reacts with polyacrylic acid to form a hard cement.[5] The poor physical properties related to conventional GICs, such as brittleness and moisture sensitivity motivated the development of improved glass ionomer cements.[6] In the 1980s, the resin-modified glass ionomer cement (RMGIC) was launched aiming to overcome the moisture sensitivity and low mechanical strength associated with the conventional glass ionomer.[7, 8] A water-soluble resin monomer (e.g., hydroxyethyl methacrylate (HEMA)) was added to the conventional GIC with an initiator, to produce a polymerization reaction in the RMGIC.[2, 8] Resin-modified glass ionomer cement (RMGIC) has improved handling properties while retaining the advantages of conventional glass ionomer such as fluoride release, chemical adhesion to the tooth structure and thermal expansion properties similar to tooth structure.[3]

SETTING REACTION:

Glass ionomer cement (GIC) is a unique water-based tooth colored restorative material that was developed to combine favorable properties of silicate cement (e.g., fluoride release and uptake, translucency and aesthetics), and polycarboxylate cement (i.e., chemical adhesion to the inorganic portion of enamel and dentin).[9] Fluoroaluminosilicate glass reacts with polyacrylic acid to form hard cement. This reaction takes place in several stages. [5] In addition to the acid-based reaction, RMGIC exhibits a polymerization reaction. This is because RMDGIC contains a water soluble resin monomer and photo-initiator.[2, 6, 10]

DENTIN BONDING

Conventional GICs demonstrates a unique chemical bond to tooth structure. This is mainly an ionic bond between carboxyl groups from the polyacrylic acid and the calcium ions present on enamel and dentin. An ion exchange layer forms between tooth structure and cement at the tooth-cement interface.[6, 11]

This chemical ionic bond phenomenon is present in RMGIC at the ion exchange interface.[12,13] In addition to the ionic bond, several studies support the formation of a resin tag hybrid layer that adds a micro-mechanical bond to the RMGIC.[12, 14]

Since first recommended by Powist et al. polyacrylic acid has been widely used as a surface substrate conditioner that improves adhesion of both conventional and resin-modified GIC to tooth structure.[15, 16] Polyacrylic acid contains carboxyl ion groups that form hydrogen bonds which promote cleansing and wetting of the substrate.

Studies show that using polycrylic acid as dentin surface pre-treatment improves the dentin bonding of both GIC and RMGIC.[15-20] However, some studies claim it does not improve the dentin bond of the RMGIC.[21]

DENTIN BOND TESTING

Laboratory testing is often done to assess dentin bonding of restorative materials.[22,23] The dentin bond strength of RMGIC was tested in several studies using the shear bond strength method with values ranged from 5-22 MPa.[24-29] El-Askary and Nassif, studied the dentin shear bond strength of nano-filled RMGIC with the use of the novel non-rinse Ketac Nano primer and other conditioning protocols (No conditioning, Nano primer only, Ketac conditioner only, Nano primer +Ketac conditioner, Nano primer + Scotchbond etchant and Nano primer + EDTA). No bond was detected when no conditioner or only

Ketac conditioner were used. The use of the pre-conditioner on the other groups significantly increased the dentin shear bond strength of the nano-filled RMGIC (Ketac Nano).[18]

Recently microtensile bond testing was carried out in several research studies to evaluate RMGIC-dentin bond strengths.[15, 17, 19, 20, 30, 31] Microtensile bond strengths were in the range of 15-35 MPa. Cohesive failure was predominant in some studies[15, 19, 20] while others have shown more mixed and adhesive failure. [17, 30]

Yip et al. investigated the microtensile dentin bond strength of several conventional GIC. In their study more than half of the failures were either adhesive or mixed. They concluded that failures of GIC with micro-tensile bond testing do not reflect the true adhesive bond to dentin.[32]

Tanumiharja et al. compared dentin microtensile bond strengths of two RMGICs (Photac™ Fil, Fuji II™ LC) to conventional GIC (Fuji IX GP™) under different conditioning protocols. They use an "hourglass-shape" trimmed microtensile method. In their study there was no significant difference between Photac™ Fil and Fuji II™ LC when any of the dentin conditioners were used. When no conditioner was used Photac™ Fil showed greater bond strength values compared to Fuji II™ LC. Conditioning significantly improved the bond strength of Fuji II™ LC while it did not affect Photac™ Fil. They related this finding to the maleic acidic content of Photac™ Fil that may act as self dentin conditioner. Most of the failures in their study were cohesive cement failure. The high cohesive failure in their study was related to the porosity of the materials used.[15]

In another study Cardoso et al. examine the microtensile dentin bonding of RMGI (Fuji II™ LC) bonded to differently prepared substrates (bur-cut, fractured, laser-irradiated)

with or without polyacrylic conditioning. The conditioning improved the microtensile bonding of Fuji II™ LC to bur-cut dentin. Total adhesive failure was observed when no conditioner was used, compared to 40% adhesive and 44% mixed with conditioner for the bur-cut group. They concluded from their study that polyacrylic acid conditioning remains important for RMGIC dentin bonding.[17]

Jordehi et al. compared the microtensile dentin bonding of laser prepared and bur-cut substrates with different GIC and RMGIC materials (Fuji II™, Vitremer, and Fuji II™ LC). Fuji II™ LC was examined with or without polyacrylic acid conditioning. Among the examined materials, the Fuji II™ LC bur-cut with conditioning group showed the highest dentin bond strengths followed by the Fuji II™ LC bur-cut non-conditioned group. For the bur-cut Fuji II™ LC groups failure was mostly cohesive.[19]

Burrow et al. examined the microtensile bond strength of a GIC (Fuji IX) and a RMGIC (Fuji II™ LC) compared to two resin adhesives (Prime & Bond and Single Bond) for both primary and permanent dentin. There was no difference in bonding values between Fuji II™ LC and the resin groups for permanent dentin. Fuji II™ LC showed mostly cohesive failure compared to mostly adhesive failure for resin groups on permanent dentin.[20]

Marquezan et al. studied the effect of bond degradation on dentin microtensile bond strength of two RMGIC materials (Fuji II™ LC and Vitremer) for both primary and permanent dentin. For the non-chemically degraded groups there was no difference between the two materials. Both show no adhesive and mostly mixed failure for the same groups.[30]

Coutinho et al. studied the microtensile enamel and dentin bond of the new nano-filled RMGC (Ketac Nano) with and without the use of the novel non-rinse Ketac Nano primer compared to conventional GIC (Fuji IX) and traditional RMGIC (Fuji II™ LC). When Ketac Nano primer was used, the nano-filled RMGIC (Ketac Nano) bonded as effectively to enamel and dentin as the conventional GIC. In contrast, it showed less bond effectiveness to both enamel and dentin when compared to the traditional RMGIC. The nano-filled RMGIC had significantly lower microtensile bond strength to both enamel and dentin when the substrate did not receive any pre-treatment. They also studied the interfacial interaction of these materials to both enamel and dentin using transmission electron microscopy (TEM). In contrast to both traditional GIC and RMGIC which showed both chemical and micro-mechanical interlocking bonds to the substrate, the nano-filled RMGIC showed mainly chemical bonding through its acrylic/itaconic acid copolymer. Its micromechanical bond is limited to the surface roughness induced by surface preparation. The nano-filled RMGIC showed no evidence of demineralization and hybridization of the surface substrate and had higher adhesive failure than the control groups.[33]

MICRO TENSILE BOND TEST

Microtensile bond strength testing was first introduce to dentistry by Sano et al. in 1994.[34] This test includes trimming the test specimens to a very small “dumbbell-shaped” or “hourglass-shaped” area about 1 mm^2 in cross-section. This “trimmed” micro tensile method has the advantage of better stress test concentration at the bonding interfacial area, but is not suitable for materials with low bond strength to tooth structure (< 5 MPa)

since trimming forces during specimen preparation may induce defects at the bonding interface. These defects initiate crack propagation that cause specimens to fail prior to testing or fail at lower bond strength.[35 32 36]

To overcome this problem a "non-trimmed" microtensile test method was developed.[37] This test includes several small beam preparations from the same sample measuring ≤ 1 mm^2 in cross-section. Microtensile bond strength testing has several advantages when compared to shear bond strength testing including: [34 35, 36, 38, 39]

- The use of fewer teeth per test.
- Smaller surface area that results in better stress distribution at the bonding interface.
- Easier to perform scanning electron microscopy ((SEM) due to the small surface area.
- Less affected by surface flaws.
- Higher bond strength values.
- Less cohesive failure.

On the other hand, this technique is very laborious, lacks agreement on guidelines, needs special preparation equipment, and may promote the dehydration of the small beams.[36 38]

Compared to the "trimmed" method the "non-trimmed" micro-tensile method is faster and less skill dependent.[35] The micro-tensile bond strength can be calculated by dividing the tensile failure load by the cross-sectional interface area.[38]

The micro-tensile testing is considered unsuitable for enamel and other brittle material (i.e. Ceramic) bond testing, since enamel shows lower bond strengths compared to dentin bonding which is contrary to what has been shown in other laboratory and clinical studies.[36] This is mainly because of the brittleness of enamel and its isotropic nature which increases the pre-test stress during sample preparation.[36]

OBJECTIVE

The purpose of this study was to investigate whether the use of novel non-rinse conditioners as substrate pre-treatment and the new paste/paste RMGIC would affect the microtensile dentin bond strength of the material in comparison to the traditional resin-modified glass ionomer cement with polyacrylic acid as a surface substrate pre-treatment.

HYPOTHESES

Null hypothesis

There will be no significant difference in microtensile dentin bond strength between the new paste/paste resin-modified glass ionomer and the powder/liquid resin-modified glass ionomer produced by the same manufacturer when the substrates are conditioned with the new “non-rinse” conditioner or polyacrylic acid.

Alternative hypothesis

The new paste/paste resin-modified glass ionomer and the powder/liquid resin-modified glass ionomer would present different microtensile dentin bond strength values when the substrates are conditioned with the new “non-rinse” conditioner than the polyacrylic acid.

MATERIALS AND METHODS

The microtensile bond strengths of four different RMGIC materials bonded to dentin following two different dentin conditioning protocols were tested (TABLE 1). Ninety-six newly extracted (within the past 6 months) non-restored human molar teeth were collected under an IUPUI/ Clarian IRB approved protocols (1106006167). The occlusal surface of the crown of each tooth was ground to expose dentin using a wheel polishing machine with 180-silicon carbide paper (300 rpm). The absence of enamel was verified using a stereomicroscope (45×). The exposed dentin was wet-finished using the same wheel polishing machine with 400 and 600-silicon carbide paper (300 rpm) to produce a standardized smear layer. Samples were stored in distilled water and then randomly allocated into eight groups (n=12) (TABLE 2).

Specimens for each of the test groups were fabricated in the sequence from 1 to 8 by conditioning the dentin surface with the respective conditioner. Then, using a clear matrix band around the circumference of the tooth, the appropriate RMGIC was placed in 2 mm-high increments and light-cured using an Optilux 400 light cure unit (Demetron Research Corp, Danbury, CT) until the glass ionomer on top of the tooth reached 5 mm-high. The output of the curing light was monitored using a Demetron radiometer (model 100, Demetron Research Corp) to maintain a >600 mW/cm^2 light output. For groups 1, 3, 4, 5, 7, and 8 the dentin was conditioned according to manufacturer's instructions. For groups 2, and 6, the dentin was conditioned with polyacrylic acid following the manufacturer's instructions for placing traditional RMGIC.

Group 1: the Ketac™ Nano primer was applied according to manufacturer's instructions to the finished dentin surface for 15 seconds using a flexible disposable applicator (Kerr

Applicators™). Replenishing was done as needed to ensure surfaces were kept wet with the primer for the recommended application time. The primer was air-dried using an air syringe for 10 seconds resulting in a dentin surface with a shiny appearance. The primed surface was light-cured for 10 seconds using the previously mentioned light curing unit. A clear Mylar matrix band was placed around the circumference of the tooth and Ketac™ Nano (shade A2) from a quick mix capsule was applied in 2 mm increments and light cured for 20 seconds per increment until an occlusal restoration depth of 5 mm was achieved.

Group 2: Ketac™ Conditioner (25% Polyacrylic acid) was applied to the polished dentin surface using a flexible disposable applicator (Kerr Applicators™). The conditioner was left on the tooth for 10 seconds, then rinsed with water spray for 10 seconds. Excess moisture was blot-dried with Kim Wipes®.

Ketac™ Nano (shade A2) was applied according to manufacturer's instructions as previously described in group 1.

Group 3: Ketac™ Nano primer was applied according to manufacturer's instructions as previously described in group 1.

Photac™ Fil (shade A2) capsules were activated using a 3M ESPE capsule activator for 2 seconds, then mixed for 15 seconds at a speed of 4300 cpm (cycle per minutes). With a clear matrix band around the circumference of the tooth, the material was placed on the tooth in 2 mm increments and light-cured for 20 seconds for each increment until a filling height of 5 mm was obtained.

Group 4: Ketac™ Conditioner (25% Polyacrylic acid) was applied according to manufacturer's instructions as previously described in group 2. Photac™ Fil (shade A2)

was then applied according to manufacturer's instructions as previously described in group 3.

Group 5: G C Self Conditioner was applied according to manufacturer's instructions to the finished dentin surface using a flexible disposable applicator (Kerr Applicators™). The conditioner was left undisturbed for 10 seconds (non-rinse conditioner). Fuji Filling™ LC (shade A2) was then applied according to manufacturer's instructions. This paste-paste product is supplied in a special "Paste Pak" cartridge that dispenses paste A and paste B in a 2:1 volume ratio (3.3 g: 1.0 g). The material was extruded onto a mixing pad and hand-mixed with a plastic spatula using overlapping strokes for 10 seconds. A clear matrix was placed around the circumference of the tooth and the mixed material was applied to the conditioned dentin surface in 2 mm increments and light cured for 20 seconds per increment until an occlusal height of 5 mm was achieved.

Group 6: GC Cavity Conditioner (20% Polyacrylic acid) was applied to the dentin surface using a flexible disposable applicator (Kerr Applicators™). The conditioner was left on the dentin surface for 10 seconds, rinsed away with a 10 second water spray, and then excess moisture was blot-dried with Kim Wipes®.

Fuji Filling™ LC (shade A2) then was also applied according to manufacturer's instructions as previously described in group 5.

Group7: GC Self Conditioner was applied according to manufacturer's instructions as previously prescribed in group 5.

Fuji II™ LC (shade A2) capsules were shaken and activated per manufacturer's instructions and mixed for 10 seconds at 4300 cpm (cycle per minuets). With a clear matrix band around the circumference of the tooth, the material was placed in 2 mm

increments and light-cured for 20 seconds for each increment until an occlusal height of 5 mm was achieved.

Group 8: GC Cavity Conditioner was applied according to manufacturer's instructions as previously described in group 6.

Fuji II™ LC (shade A2) then was also applied according to manufacturer's instructions as previously explained in group 7.

All specimens were stored at 37 °C for 24 hours in 100% humidity before cutting to obtain the non-trimmed beams to be used for the microtensile bond testing.

Microtensile bond testing:

Using a low-speed saw with a diamond blade (Isomet, Buehler, Lake Bluff, IL, USA), (Figure 1) each specimen was vertically sectioned into serial slabs and the slabs sectioned further into beams with a cross-sectional area of approximately 0.8×0.8 mm^2 (Figure 2). Nine beams were used from each specimen. Each beam was attached to a modified Bencor Multi-T testing apparatus (Danville Engineering Co., Danville, CA, USA) using a cyanoacrylate-based adhesive (Zapit, Dental Ventures of America Inc., Corona, CA, USA) (Figure 3) and stressed to failure in tension using an universal testing machine (MTS Sintech Renew 1123, Eden Prairie, MN, USA) at a cross-head speed of 1 mm/min (Figure 4).

Failure mode examination:

Debonded specimens were examined under a stereomicroscope at 45× magnification to evaluate the fracture pattern. Failure modes were classified:

- Adhesive failure at the dentin material interface.
- Cohesive failure within the dentin surface or within the material itself.
- Mixed failure partially adhesive and partially cohesive.
- Glue-side detachment from the testing jig before failure. This classification was added since several beams in each group were detached from the testing apparatus before failure.

In addition, the dentin sides of eight randomly chosen representative debonded beams were examined under a scanning electron microscope SEM (JEOL JSM-5310LV, Jeol Ltd, Tokyo, Japan). The specimens were sputter-coated with gold, then evaluated at 150×, 500× and 1500× magnification at 20 kV acceleration voltages. The qualitative information aims to correlate the failure mode result of the stereomicroscope to the randomly chosen SEM specimens.

Statistical Methods

Based on prior studies[40] the correlation among beams from the same specimen was estimated to be 0.3 and a standard deviation of 20 MPa. With a sample size of 12 specimens per group, with each specimen divided into 9 beams, the study had 80% power to detect a difference in microtensile bond strengths of 15 MPa between any two groups, assuming a 5% significance level for each test.

Comparisons between the groups for differences in microtensile peak stress were performed using a Weibull distribution survival analysis, using the stress required for failure in place of the usual “time to event” seen in typical survival analyses. The analysis included a “frailty” term to correlate the measurements from beams fabricated from the

same tooth. Differences between the groups for type of failure (adhesive vs. mixed or cohesive or glue-side) were analyzed using generalized estimating equation methodology applied to logistic regression models to handle the correlations among beams from the same specimen.

RESULTS

Microtensile Bond Strength:

The mean microtensile bond strength with standard error and the maximum and minimum values for each group are shown in Table 3. The number of teeth and beams that were tested in the study are also shown (Table 3). Figure 12 shows the mean microtensile bond strengths with standard error.

Weibull-distribution survival analysis was used to compare the differences in microtensile peak stress between the groups. The Weibull distribution survival analysis used the stress required for failure in place of the usual "time to event" seen in typical survival analyses. Figure 13 show the survival functions using the individual observations while Figure 14 shows the survival curves fitted by the Weibull models. In Figure 13 and 14, the *y* axis shows survival probability of failure from 1 to 0 where 1 no failures and 0 total failure of all the samples while the *x* axis represents microtensile peak stress of failure in MPa. For example group 1 (Ketac Nano + Ketac Nano Primer) starts to failed at low stress compared to group 3 (Photac Fil + Ketac Nano Primer) or group 7 (Fuji II LC + GC Self Conditioner) which failed at higher stress values.

For Ketac Nano groups (Groups 1 and 2) there was no significant difference in microtensile bond strength when either Ketac Nano Primer or Ketac Conditioner were used as dentin surface pretreatment ($p > 0.05$). Photac Fil groups also show no significant difference in microtensile bond strength when either Ketac Nano Primer or Ketac Conditioner were used as the dentin surface pretreatment ($p > 0.05$). The mean microtensile bond strength for the Photac Fil groups was significantly higher than Ketac Nano groups when either Ketac Nano Primer or Ketac Conditioner were used ($p < 0.05$).

For Fuji Filling LC with cavity conditioner (Group 6), no bond strengths were measured since total bond failure occurred before or during sample preparation. Fuji II LC showed significantly higher mean microtensile bond strength when GC Self Conditioner was used as the dentin surface conditioner compared to GC Cavity Conditioner ($p < 0.05$). There was no significant difference between Fuji Filling LC with GC Self Conditioner and the Fuji II LC groups ($p > 0.05$).

There was no significant difference between Photac Fil with Ketac Nano Primer and Fuji II LC with GC Self Conditioner ($p > 0.05$). These two groups showed the highest mean microtensile bond strengths (20.0 ± 1 MPa) of all of the groups. Ketac Nano with Ketac Nano Primer showed the lowest mean microtensile bond strength (9.5 ± 1 MPa) followed by Ketac Nano with Ketac Conditioner (11.0 ± 1 MPa), Fuji II LC with GC Cavity Conditioner (14.1 ± 0.9 MPa), Fuji Filling LC with GC Self Conditioner (15.1±1 MPa) and Photac Fil with Ketac Conditioner (16.8 ± 1 MPa).

Failure mode:

Failure mode analyses for all the study groups are shown in Figure 15 and Table 4. There was no significant difference in failure mode between the Ketac Nano groups when either Ketac Nano Primer or Ketac Conditioner were used as the dentin surface pretreatment ($p > 0.05$). Photac Fil with Ketac Conditioner had significantly more adhesive failure than Photac Fil with Ketac Nano Primer ($p < 0.05$).

Fuji II LC with GC Cavity Conditioner had significantly more adhesive failure than Fuji II LC with GC Self Conditioner or Fuji Filling LC with GC Self Conditioner ($p < 0.05$).

Fuji II LC with GC Cavity Conditioner and Photac Fil with Ketac Conditioner had the highest adhesive failure mode among all the other groups, 86% and 82% respectively. There was no significant difference between these two groups ($p > 0.05$). Fuji Filling LC with GC Self Conditioner had the lowest adhesive failure (43%) followed by Fuji II LC with GC Self Conditioner (49%), Photac Fil with Ketac Nano Primer (64%), Ketac Nano with Ketac Nano Primer (69%) and Ketac Nano with Ketac Conditioner (79%).

SEM results:

Figures (5-11) shows the SEM results and image descriptions of the dentin side for seven randomly selected failed beams. There was no SEM examination for group 6 since it had total pre-test failures. For figures (5, 6, 7, 8 and 10) that represent Group (1, 2, 3, 4 and 7) respectively, SEM result shows mixed type failure while the stereomicroscope showed apparent adhesive failures for these beams. The SEM of Group 5 (Figure 9) shows cohesive type failure while the stereomicroscope showed apparent adhesive failures for those beams. The only agreement between SEM and stereomicroscope results was with Group 8 (Figures 11) where both methods show cohesive failure.

FIGURES

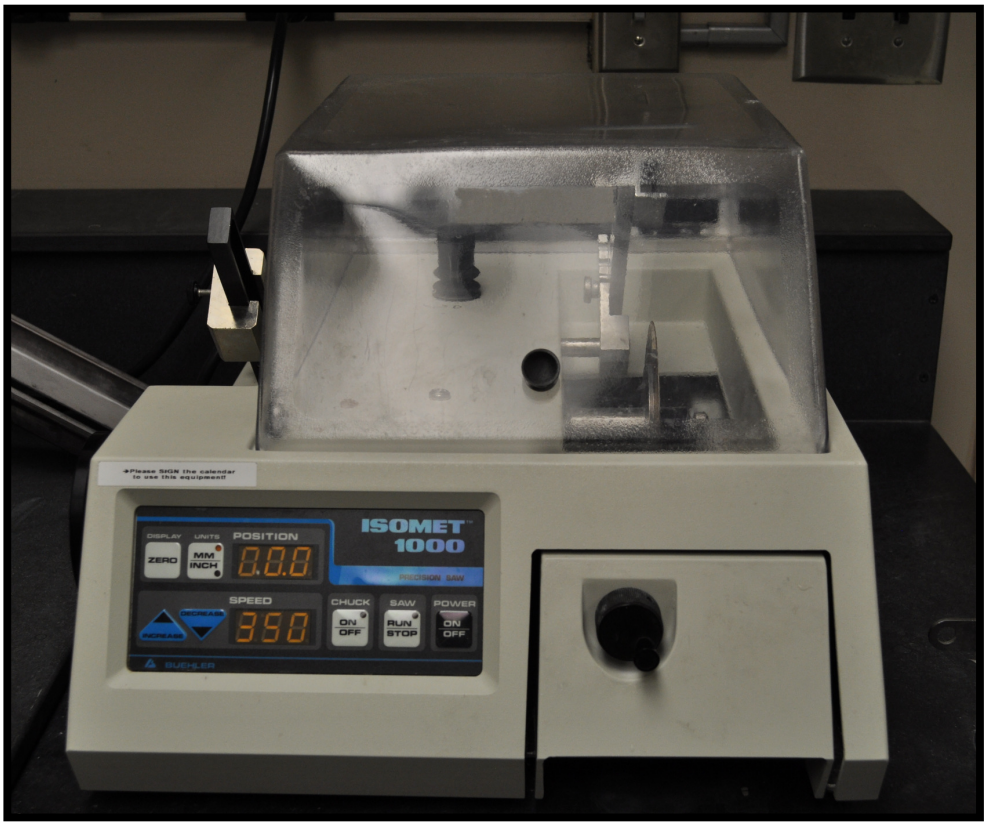

Figure 1. Isomet 1000, Buehler, Lake Bluff, IL, USA.

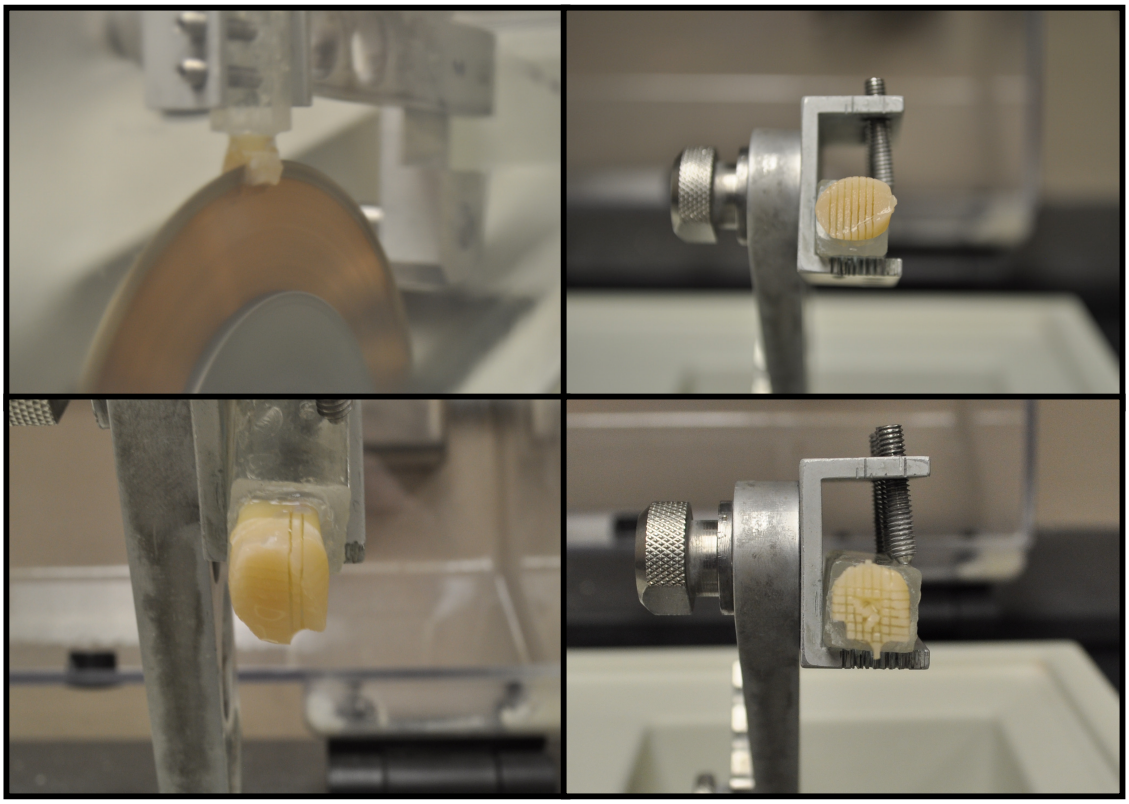

Figure 2. Beams sectioning.

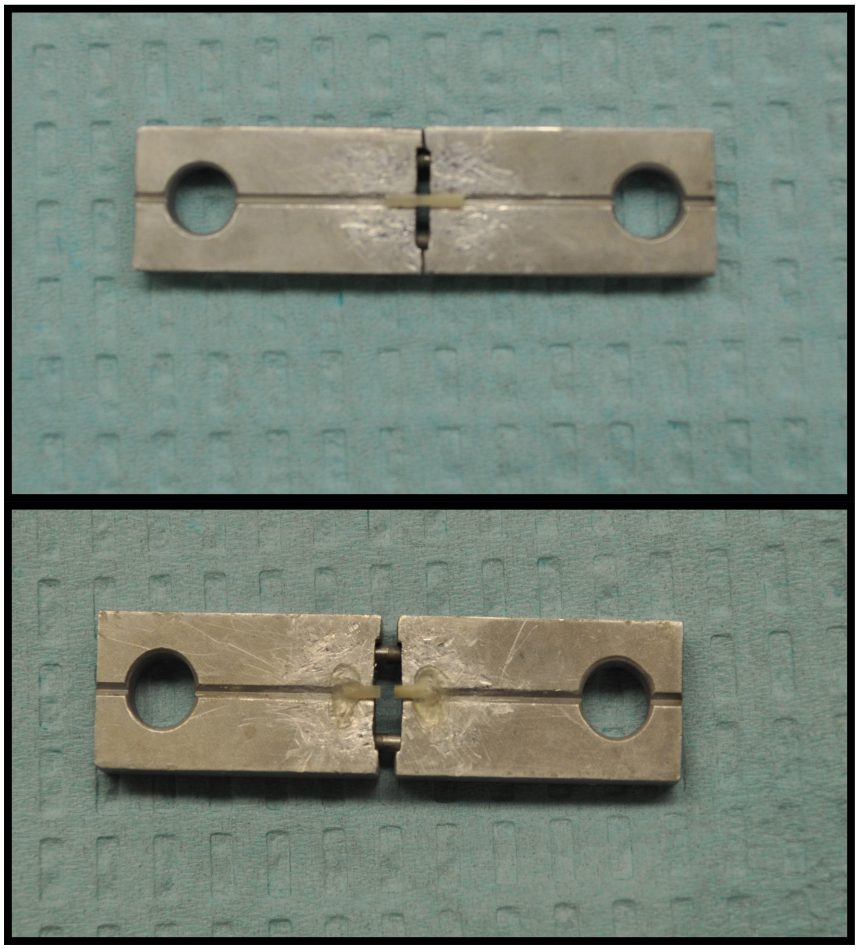

Figure 3. Microtensile beams before and after testing.

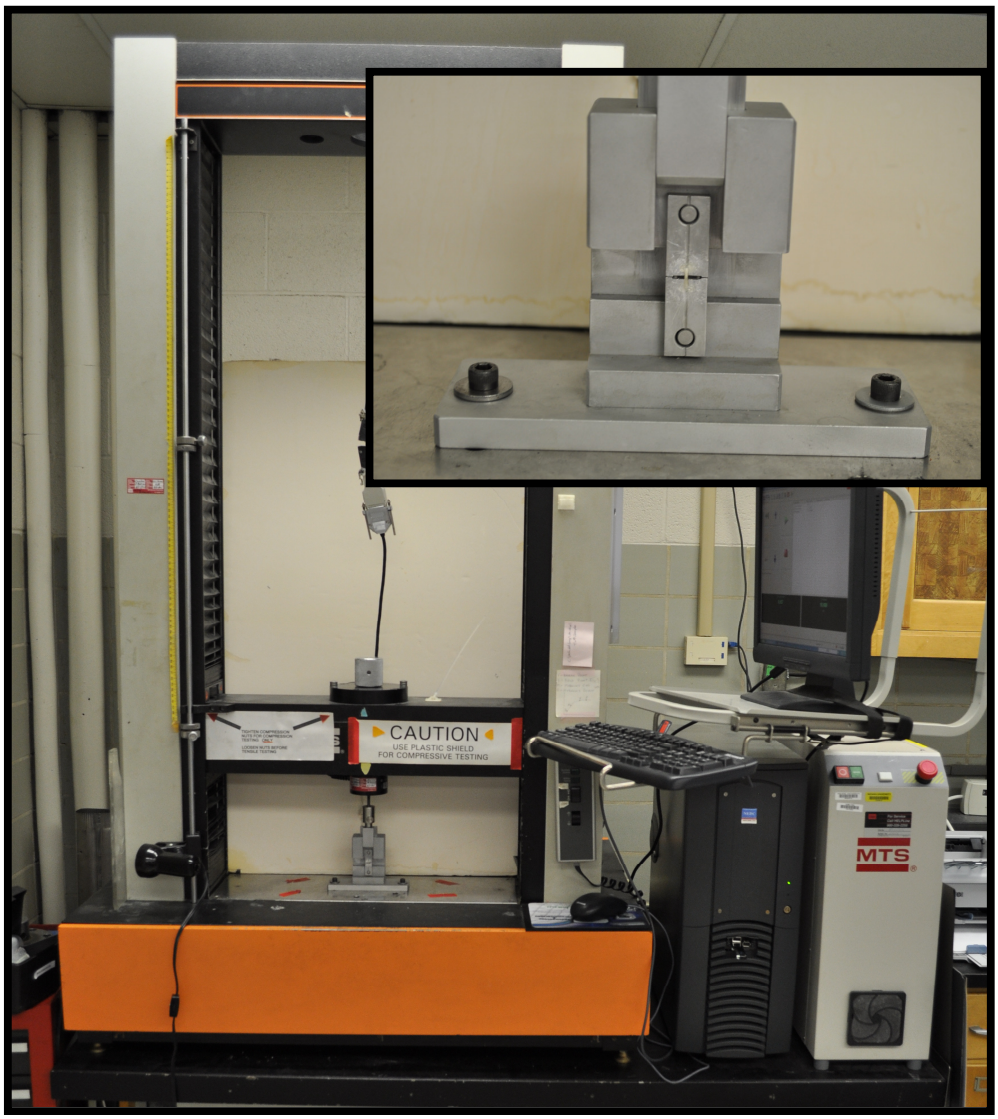

Figure 4. Universal testing machine (MTS Sintech Renew 1123, Eden Prairie, MN, USA).

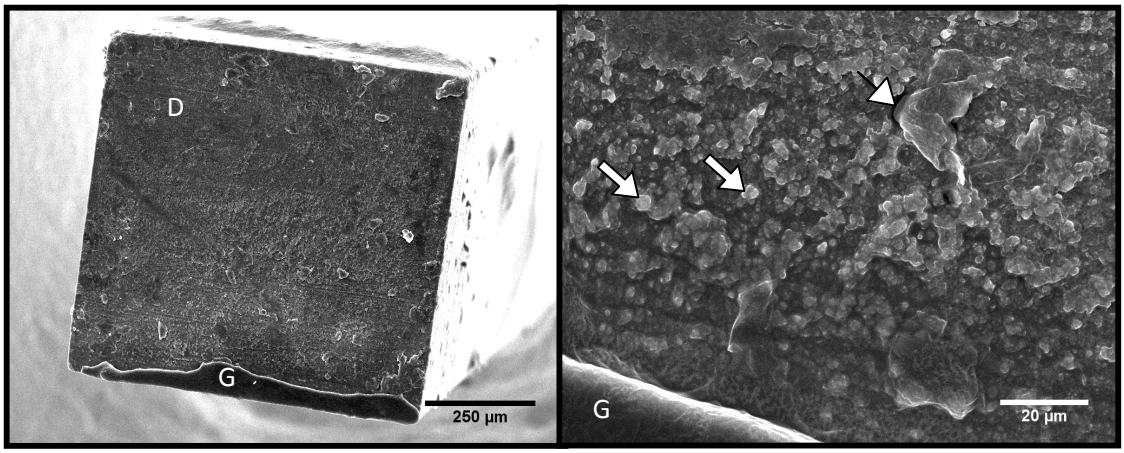

Figure 5: SEM images of apparently adhesive failure under light microscopy; SEM images show a mixed type of failure where both dentin (D) and RMGI (G) can be identified on the fractured dentin surface of the beam. At higher magnification, SEM image clearly shows interfacial dentin surface covered by remnants of RMGI and Nano filler particles (arrow) occlude dentinal tubule (arrow head). (Group 1: Ketac™ Nano + Ketac™ Nano Primer) (150 x and 1500 x).

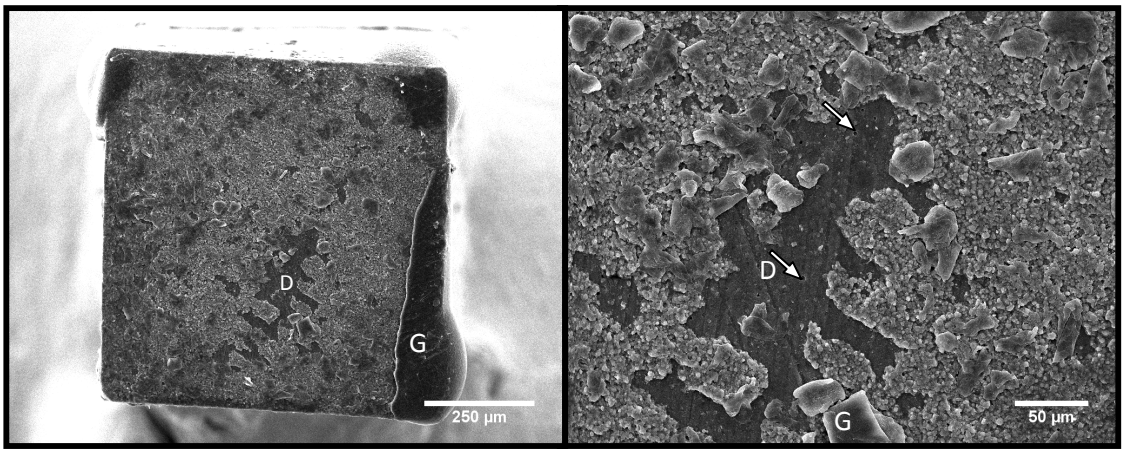

Figure 6: SEM images of apparently adhesive failure under light microscopy; SEM images show a mixed type of failure where both dentin (D) and RMGI (G) can be identified on the fractured dentin surface of the beam. The higher magnification SEM shows interfacial dentine surface covered by remnants of RMGI (G) and Nano filler particles, several open dentinal tubules can also be identified (arrows). (Group 2: Ketac™ Nano + Ketac™ Conditioner) (150 x and 500 x).

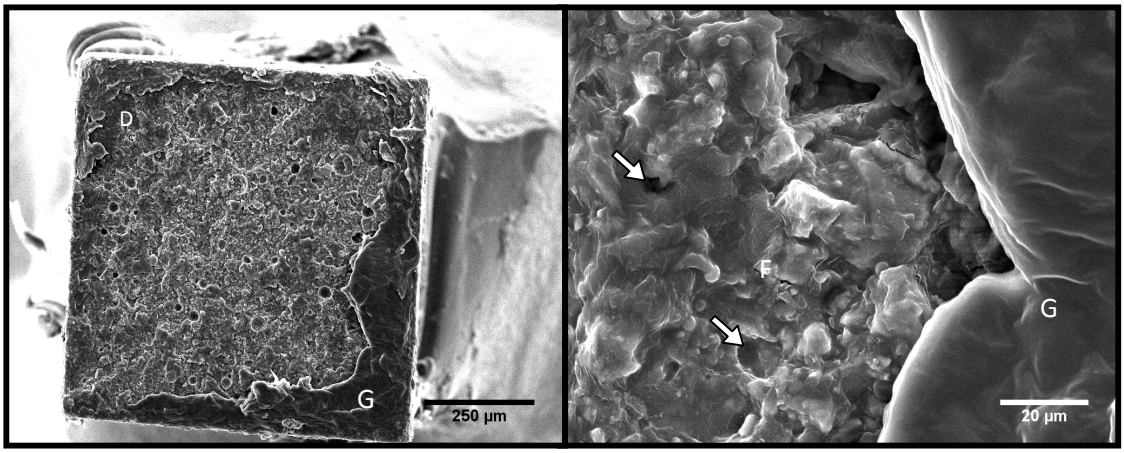

Figure 7: SEM images of apparently adhesive failure under light microscopy; SEM images show a mixed type of failure where both dentin (D) and RMGI (G) can be identified on the fractured dentin surface of the beam (F) shows several air bubbles. The higher magnification SEM shows interfacial dentine surface (F) totally covered by remnants of RMGI (G) and air bubbles (arrows) no dentin surface can be identified. (Group 3: Photac™ Fil + Ketac™ Nano Primer) (150 x and 1500 x).

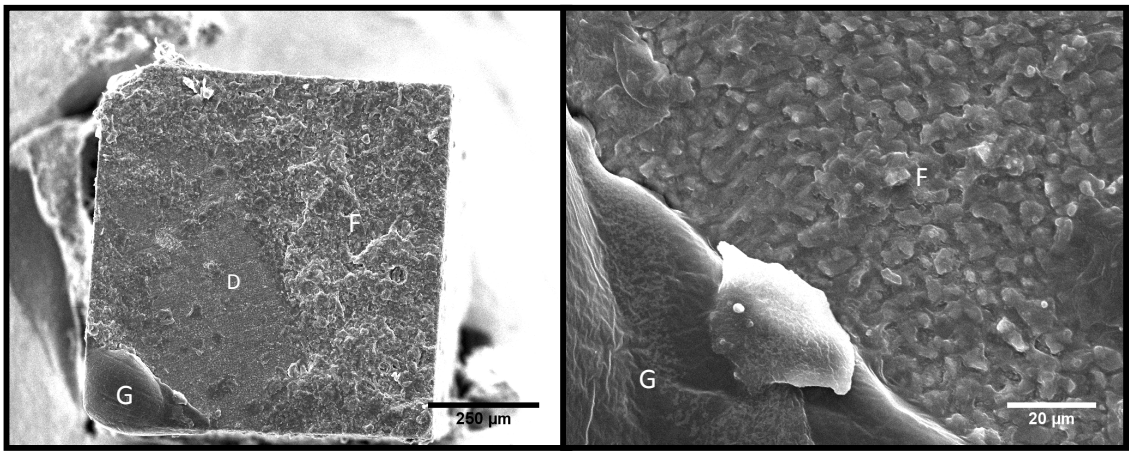

Figure 8: SEM images of apparently adhesive failure under light microscopy; SEM images show a mixed type of failure where both dentin (D) and RMGI (G) can be identified on the fractured dentin surface of the beam. The higher magnification SEM shows interfacial dentine surface (F) covered by remnants of RMGI (G). (Group 4: Photac™ Fil + Ketac™ Conditioner) (150 x and 1500 x).

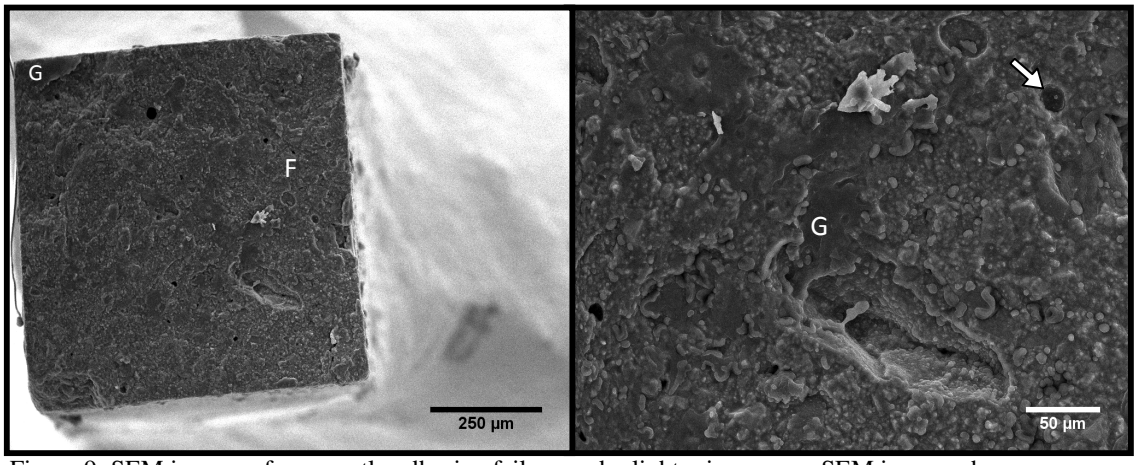

Figure 9: SEM images of apparently adhesive failure under light microscopy; SEM images show a cohesive type of failure where the fracture dentine surface of the tested beam is totally covered by RMGI (G). The higher magnification SEM shows interfacial dentine surface (F) covered by remnants of RMGI (G), some air bubbles can be identified (arrow). (Group 5: Fuji Filling™ LC + G C Self Conditioner) (150 x and 500 x).

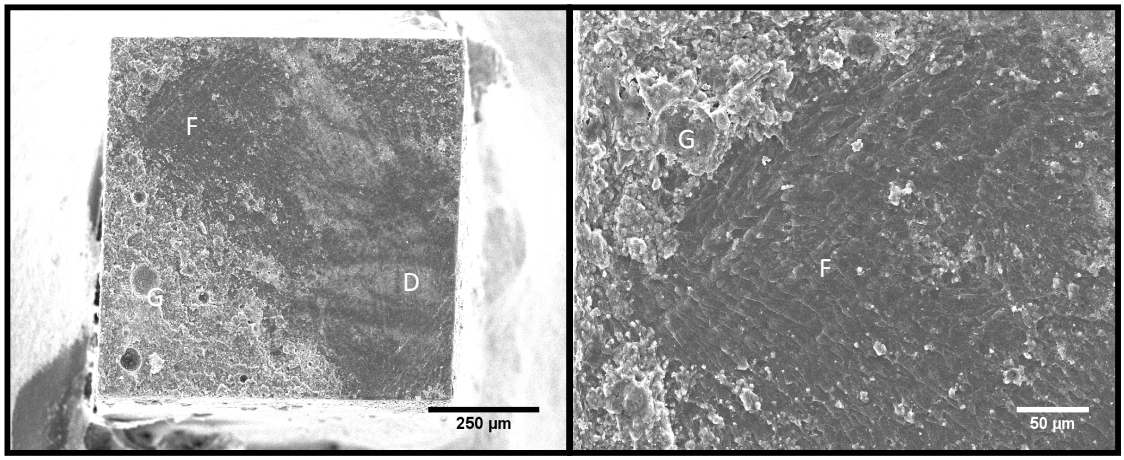

Figure 10: SEM images of apparently adhesive failure under light microscopy; SEM images show a mixed type of failure where both dentin (D) and RMGI (G) can be identified on the fractured dentin surface of the beam. The higher magnification SEM shows interfacial dentine surface (F) covered by remnants of RMGI (G). (Group 7: Fuji II™ LC + G C Self Conditioner) (150 x and 500 x).

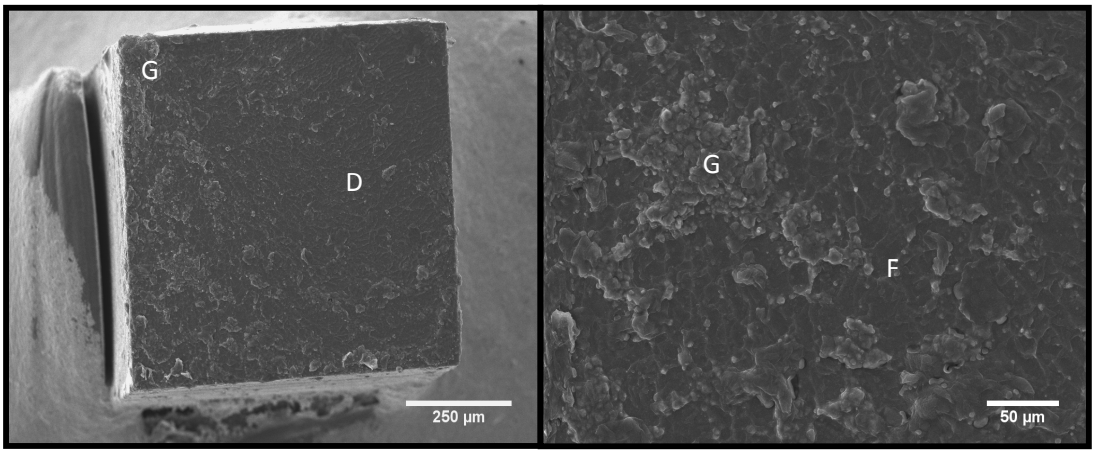

Figure 11: SEM images of apparently mixed failure under light microscopy; SEM images show a mixed type of failure where both dentin (D) and RMGI (G) can be identified on the fractured dentin surface of the beam. The higher magnification SEM shows interfacial dentine surface (F) covered by remnants of RMGI (G). (Group 8: Fuji II™ LC + GC Cavity Conditioner) (150 x and 1500 x).

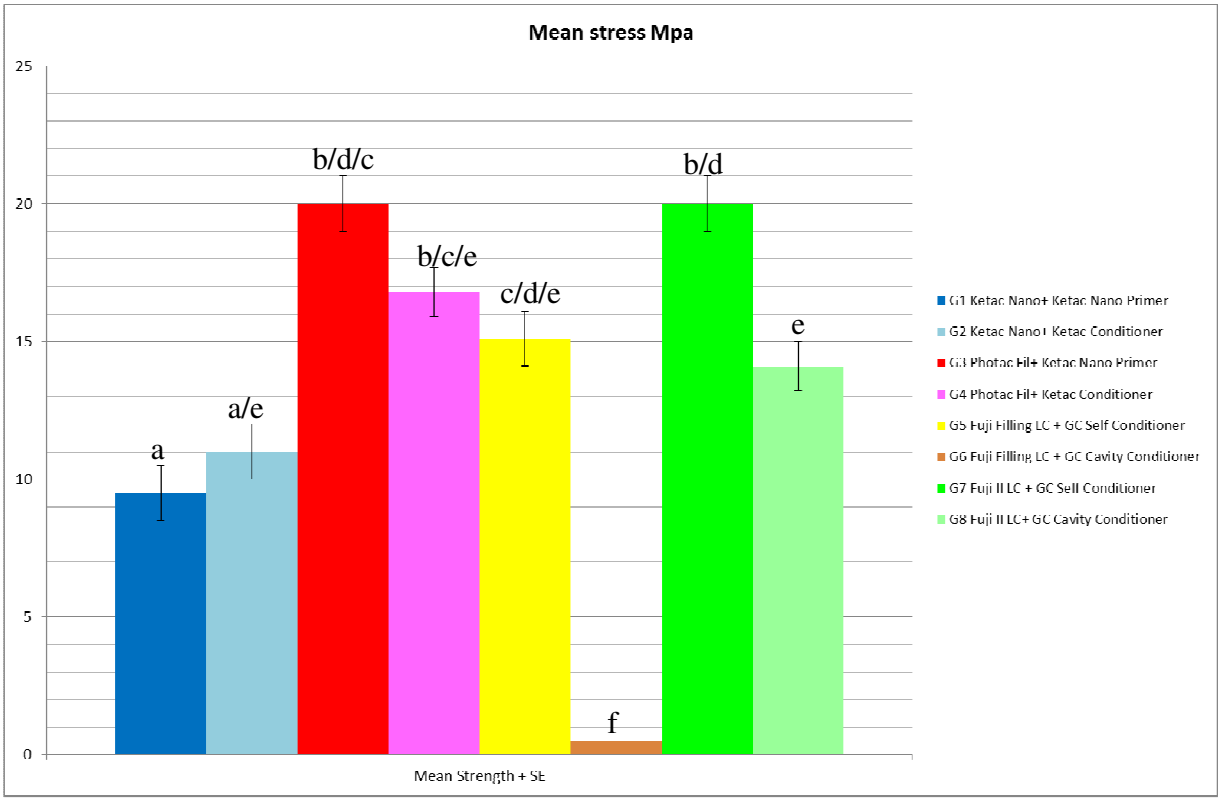

Figure 12. Mean Strength with standard error.

*Materials with one or more same letter are statistically not significant ($p > 0.05$).

*Group 6 mean strength = zero there was no bond

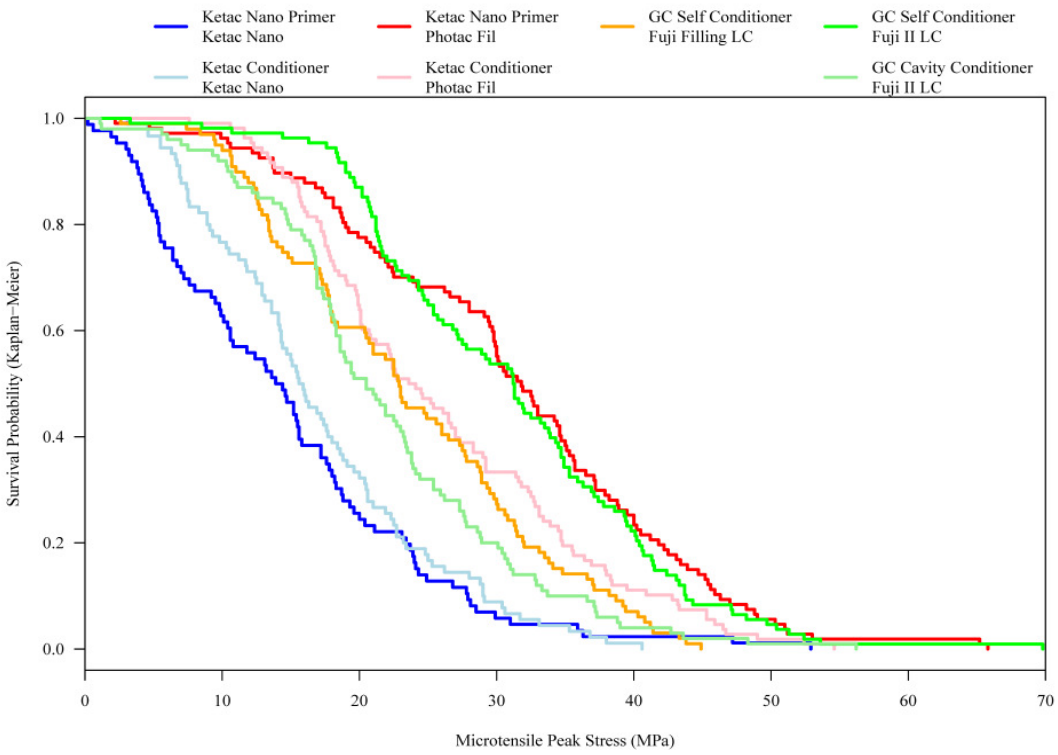

Figure 13: The survival functions using the individual observations.

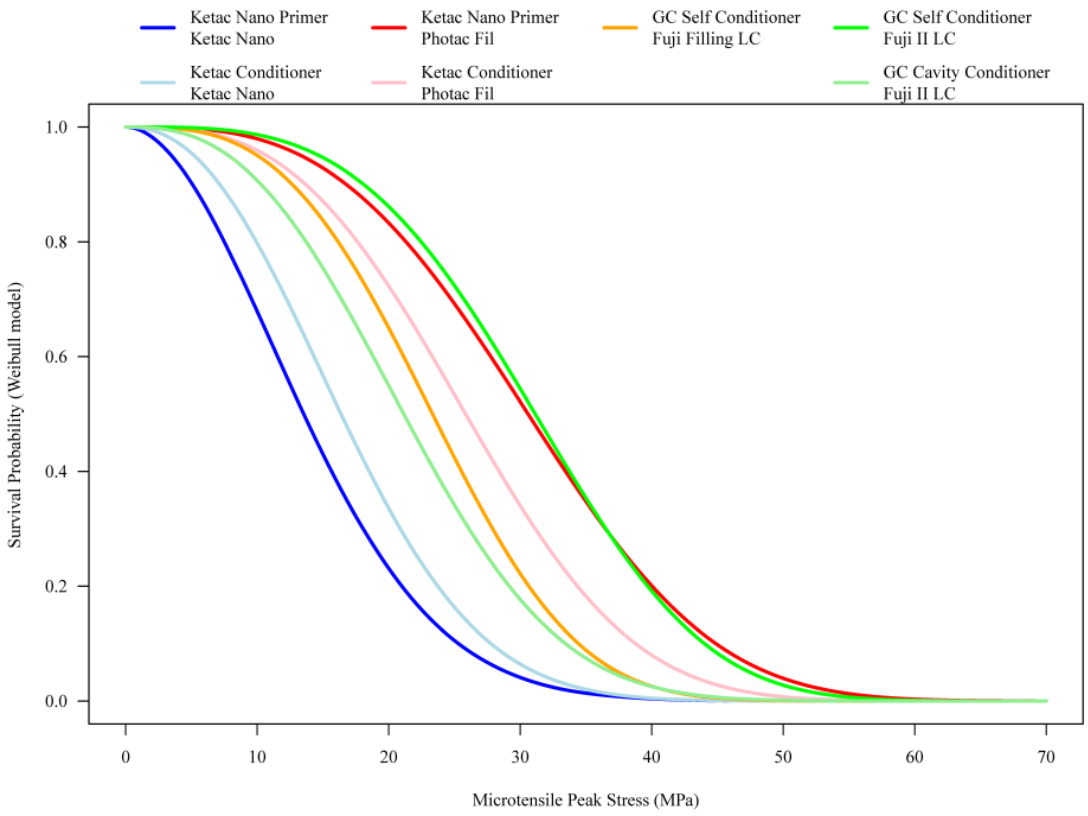

Figure 14: The survival curves fitted by the Weibull models.

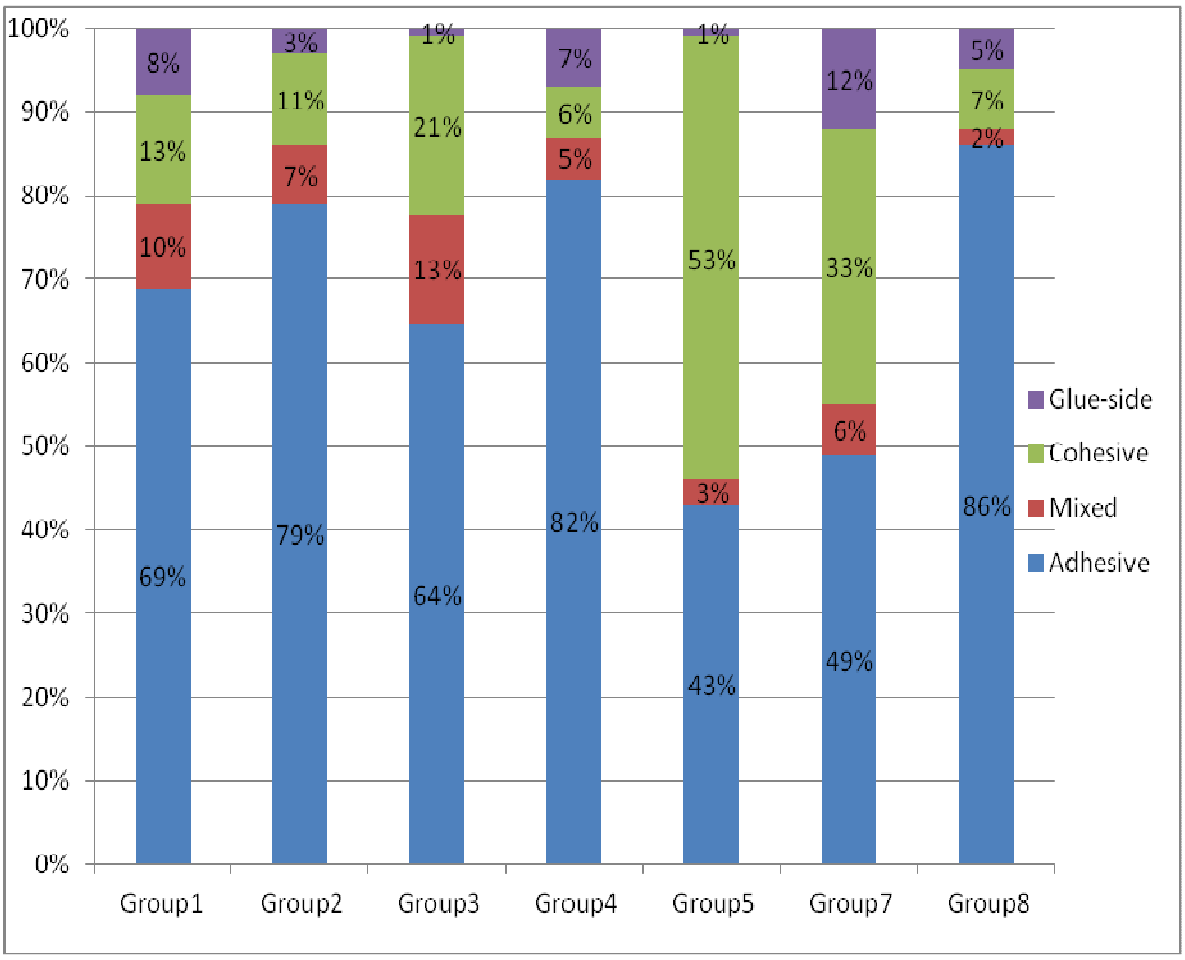

Figure 15: Failure mode in percentage (%).

*Group 6 were not shown here because it had total adhesive failure, there was no bond.

TABLES

Table 1. Materials compositions.

	Batch Number	compositions
Ketac™ Nano (3M ESPE)	268895 264728	**Paste A**: 40-50% Silane treated glass20-30% Silane treated zirconia, 5-15% POLYETHYLENE GLYCOL DIMETHACRYLATE (PEGDMA), 5-15% Silane treated silica, 1 - 15% 2-HYDROXYETHYL METHACRYLATE (HEMA), < 5% Glass powder, < 5% BISPHENOL A DIGLYCIDYL ETHER DIMETHACRYLATE (BISGMA) , < 1% TRIETHYLENE GLYCOL DIMETHACRYLATE (TEGDMA). **Paste B**: 40 - 60% Silane treated ceramic, 20 - 30% copolymer of acrylic and itaconic acids, 10 - 20% water, 1 - 10% HEMA.
Ketac™ Nano Primer (3M ESPE)	N253374 N241199	40 - 50 % Water, 35- 45 % HEMA, 10 - 15% copolymer of acrylic and itaconic acids
Photac™ Fil (3M ESPE)	439731 440857	L: 30 - 50% Polyethylenepolycarbonicacid, 25 - 50% HEMA, 20 - 30% Water, DIURETHANE DIMETHACRYLATE 3 - 10%, 5 - 10% Magnesiumhema Ester . P: > 99% Glass powder.
Ketac™ Conditioner (3M ESPE)	431890 405279	70-80% water, 20-30% Polyacrylic acid.
Fuji Filling™ LC (G C America)	1010061 1011251	**Paste A:** 75-85% Alumino-silicate glass, 10-12% HEMA,2-5%Urethanedimethacrylate **Paste B:** 20-30% Distilled water, 20-30% Polyacrylic acid, 12-15% Urethanedimethacrylate, 10-15% Silicone dioxide.
G C Self Conditioner (GC America)	1011161	28-40% Ethanol, 30-35% Distilled water, 20-30% HEMA, 5-10% 4-Methacryloxyethyltrimellitate anhydride.
Fuji II™ LC (G C America)	1009221	P: 100% Alumino-silicate glass. L: 20 - 30% Distilled water, 20 - 30%Polyacrylic acid, 30 - 35% HEMA, <10% Urethanedimethacrylate, <1% Camphorqunone
GC Cavity Conditioner (GC America)	1103151	20 %Polyacrylic acid, 77% Distilled water , 3%Aluminum chloride hydrate, <0.1%Food additive Blue No. 1.

Table 2. Experimental groups.

GROUPS (N=12)	MATERIAL AND MANUFACTURER	PRETREATMENT
1	Ketac™ Nano (3M ESPE)	Ketac™ Nano Primer (3M ESPE)
2	Ketac™ Nano (3M ESPE)	Ketac™ Conditioner (3M ESPE) (Polyacrylic acid)
3	Photac™ Fil (3M ESPE)	Ketac™ Nano Primer (3M ESPE)
4	Photac™ Fil (3M ESPE)	Ketac™ Conditioner (3M ESPE) (Polyacrylic acid)
5	Fuji Filling™ LC (GC America)	GC Self Conditioner (GCAmerica)
6	Fuji Filling™ LC (GC America)	GC Cavity Conditioner (GC America) (Polyacrylic acid)
7	Fuji II™ LC (GC America)	GC Self Conditioner (GCAmerica)
8	Fuji II™ LC (GC America)	GC Cavity Conditioner (GCAmerica) (Polyacrylic acid)

Table 1. Peak Stress (MPa).

Group	N Teeth	N Beams	Min	Max	Mean (SE)	Weibull Characteristic Strength	Weibull Modulus
1	10	84	0.2	33.8	9.5 (1.0)	16.4	1.9
2	10	89	2.0	26.0	11.0 (1.0)	19.3	2.3
3	12	106	1.4	42.1	20.0 (1.0)	34.7	3.1
4	11	107	4.9	34.9	16.8 (0.9)	29.3	3.0
5	12	98	1.7	28.7	15.1 (1.0)	26.3	3.1
7	12	107	2.1	44.7	20.0 (1.0)	34.7	3.5
8	12	99	0.8	36.0	14.1 (0.9)	24.4	2.6

Table 2. Failure Mode, N beams (%).

Mode / Group	Adhesive	Mixed	Cohesive	Glue-side
1	59 (69%)	9 (10%)	11 (13%)	7 (8%)
2	71 (79%)	6 (7%)	10 (11%)	3 (3%)
3	69 (64%)	14 (13%)	23 (21%)	1 (1%)
4	89 (82%)	5 (5%)	6 (6%)	8 (7%)
5	43 (43%)	3 (3%)	52 (53%)	1 (1%)
7	53 (49%)	6 (6%)	36 (33%)	13 (12%)
8	86 (86%)	2 (2%)	7 (7%)	5 (5%)

DISCUSSION

Microtensile bond strength testing was first introduced to dentistry by Sano et al, in 1994.[34] The early method prescribed by Sano includes trimming forces to prep the sample for micro tensile testing. Several studies did not recommend this method for testing low bonded materials (< 5MPa) since frequent pre-test failures are associated with this method.[36] The non-trimmed method recently was developed to overcome this problem associated with such materials.[37] In this study, the non-trimmed microtensile method was used. We were able to test a very low microtensile bond value as low as (0.2 MPa). Other than group 6 where all samples failed during specimen preparation, the number of the pre-test failures in our study was very low (5 teeth totally debonded and 30 individual beams failed among the various groups). The pre-test failures were not included in our statistical analysis.

Other advantages of microtensile bond strength testing include: the use of fewer teeth per test; a smaller surface area that results in better stress distribution at the bond interface; easier to perform a scanning electron microscope (SEM) examination, less effect of surface flaws; higher bond strength value; and less cohesive failure.[34 35, 36, 38, 39] The results of our study indicate a low cohesive failure of 20.77 % of all the tested samples. The mean of the microtensile bond strength in this study was (9.5-20 MPa) which is consistent with other studies. [15, 17, 19, 20, 30, 31]

On the other hand, this technique is very laborious, lacks accepted guide lines, requires special preparation equipment, and may promote the dehydration of the small beams.[36 38] To minimize the beam dehydration in this study the beams for each tooth were tested immediately after preparation.

Since first recommended by Powist et al, polyacrylic acid has been widely used as a surface substrate conditioner that improves adhesion of both conventional and resin-modified GIC to tooth structure.[15, 16]

In the current study, the use of the new Ketac Nano primer with the new paste/paste RMGIC (Ketac Nano) did not improve the microtensile dentin bond strength in comparison to the use of polyacrylic acid as conditioner for the dentin substrate. ` This finding is similar to what Coutinho et al. had found where Ketac Nano had inferior dentin microtensile bond compared to traditional RMGIC.[33] Though there was no statistical difference, the use of the new Ketac Nano primer with the traditional RMGIC (Photac Fil) showed the highest mean value compared to other materials from the same manufacturer. This finding could be related to the high content of hydroxyethyl methacrylate (HEMA) in the new Ketac Nano primer that may increased the micro-mechanical bond in addition to the chemical dentin bond (Table 1). The new paste/paste RMGIC (Ketac Nano) groups had higher incidents of pre-test failures during beam preparation where individual beams failed or the whole restoration broke especially on the second vertical cut. This may be related to the ceramic nano filler content of this material that increases the material brittleness. Brittle materials are considered unsuitable for microtensile bond testing since higher pre-test failure and lower bond values are expected.[36] This may explain the low bonding value for this material compare to other groups in this study.

The use of the new GC Self Conditioner did not improve the microtensile bond strength of the new RMGIC (Fuji Filling LC) to dentin. By contrast, the new GC Self Conditioner enhanced the dentin bond of traditional RMGIC (Fuji II LC). There was no

bond when the new RMGIC (Fuji Filling LC) was placed on polyacrylic acid conditioned dentin. This suggests that it may depend on the micro-mechanical bond of hydroxyethyl methacrylate (HEMA) in the new GC Self Conditioner rather than a chemical bond previously shown with traditional RMGIC (Fuji II LC).[13, 15] On the other hand, hydroxyethyl methacrylate (HEMA) in the new GC Self Conditioner may enhance the bond of traditional RMGIC (Fuji II LC) with additional micro-mechanical dentin bonding. This may explain the higher microtensile bond values observed for this group.

In this study debonded specimens were examined under a stereomicroscope at 45× magnification to evaluate the fracture pattern. Failure modes were classified as Adhesive (failure at the dentin material interface), Cohesive (failure within the dentin surface or within the material itself), Mixed (failure partially adhesive and partially cohesive) and Glue-side detachment from the testing jig before failure.

The current study shows mostly adhesive failure in all the groups' except group five that shows predominant cohesive failure. This result corresponds to several studies where adhesive failure was detected.[17, 30] In contrast, other studies show a predominant cohesive failure.[15, 19, 20] They related there finding of high cohesive failure to the porosity of the materials itself. In our study most of the examined groups are newly introduced materials and conditioner which are different from the materials they examined.

Several literature reviews suggest the use of higher magnification methods (i.e. SEM) to confirm the findings of the stereomicroscope.[38, 41] It is difficult to truly differentiate between the mode of failure especially adhesive and mixed failure. The term apparent

failure should be use to describe the stereomicroscope findings.[38] SEM analysis of randomly selected specimens in this study showed that all of bond failures that were classified an apparent adhesive failure under the stereomicroscope were actually mixed failures showing that the stereomicroscope is inadequate for assessing failure mode.

The use of the Weibull statistics in this study was done to provide better information regarding the probability of failure. The Weibull distribution survival analysis used the stress required for failure in place of the usual "time to event" seen in typical survival analyses. Much of the recent literature recommends the use of Weibull statistics to analyze and compare the data of microtensile bond testing.[38, 41]

SUMMARY AND CONCLUSIONS

The purpose of this study was to investigate whether the use of novel non-rinse conditioners as substrate pre-treatment with the new paste/paste RMGIC would affect the microtensile dentin bond strength of the material in comparison to the traditional resin-modified glass ionomer cement with polyacrylic acid as a surface substrate pre-treatment. Four materials were tested and compared in this study, including: Ketac™ Nano, Photac™ Fil, Fuji II™ LC and Fuji Filling™ LC. The dentin was pre-treated using one of four conditioners, including: Ketac™ Nano Primer, Ketac™ Conditioner, GC Cavity Conditioner and G C Self Conditioner. The micro-tensile dentin bond test, the failure mode analysis and SEM evaluation were used to examine these materials.

From the data collected, the results can be summarized as follows:

1) The micro-tensile bond testing may not be suitable to test the Ketac™ Nano since it has higher pre-test failure and low bond value compare to other materials when either the non-rinse conditioners or polyacrylic acid were used.
2) The Fuji Filling™ LC should only used with its self conditioner (GC Self Conditioner) since no bond was detected when polyacrylic acid was used.
3) The use of the novel non-rinse conditioners improves the dentin bond of Fuji II™ LC.

In conclusion, under the conditions of this study, the use of novel non-rinse conditioners did not improve the dentin bond of the new paste/paste RMGIC. The use of the novel non-rinse conditioners improved the dentin bond of one of the traditional RMGIC (Fuji II™ LC).

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ABSTRACT

MICROTENSILE BOND STRENGTH OF NEW PASTE/PASTE RESIN-MODIFIED GLASS IONOMER CEMENT SYSTEMS: THE EFFECT OF DENTIN PRETREATMENT

by

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Background: In order to improve the clinical performance of RMGIC, 3M ESPE and GC America introduced paste/paste resin-modified glass ionomer cements, Ketac™ Nano *and* Fuji Filling™ LC, respectively. Both companies developed non-rinse substrate conditioners (i.e., Ketac Nano Primer-3M ESPE and GC Self-Conditioner-GC America) that manufacturer's state should be used with these new materials instead of the conventional polyacrylic acid. It has been also advised by both manufacturer's to use this novel substrate conditioner with the previously marketed RMGICs. Objective: To investigate whether the use of novel non-rinse conditioners (i.e., Ketac Nano Primer 3M ESPE and GC Self Conditioner GC America) as substrate pre-treatment and the new paste/paste resin-modified glass-ionomer cement, RMGIC (*Ketac™ Nano* 3M ESPE *and* Fuji Filling™ LC GC America) would affect the microtensile dentin bond strength (μTBS) of the material when compared to the traditional RMGIC with polyacrylic acid as a surface substrate pre-treatment. Materials and Methods: 96 extracted non-restored human molars were sectioned to expose occlusal dentin. Dentin surface was finished with

SiC paper to standardize the smear layer. Bonding protocols of the different materials to dentin were performed following the use of two dentin conditioners. Eight groups (n=12) were tested: G1: Ketac Nano Primer + Ketac Nano, G2: Ketac Conditioner + Ketac Nano, G3: Ketac Nano Primer + Photac Fil, G4: Ketac Conditioner + Photac Fil, G5: GC Self Conditioner + Fuji Filling LC, G6: GC Cavity Conditioner + Fuji Filling LC, G7: GC Self Conditioner + Fuji II LC and G8: GC Cavity Conditioner + Fuji II LC. The specimens were stored in 37°C for 24h in 100% humidity before cutting non-trimmed beams for the μTBS with cross-sectional areas of approximately 0.8 × 0.8 mm^2. Nine beams were used from each specimen. Testing was done using a universal testing machine at a cross-head speed of 1mm/min. Debonded specimens were examined under a stereomicroscope at 45× magnification to evaluate the failure mode. Eight randomly chosen representative debonded beams were imaged under a scanning electron microscope (SEM). Results: μTBS in MPa (mean ± SE) were: G1: 9.5±1.0, G2: 11.0±1.0, G3:20.0±1.0, G4:16.8±0.9, G5: 15.1±1.0, G6: pre-test failure, G7: 20.0±1.0, G8:14.1±0.9. Weibull-distribution survival analysis was used to compare the differences in microtensile peak stress among the groups. Group 5 has cohesive predominant failure mod while the other groups have adhesive predominant failure. Conclusion: Within the limitations of this study, the use of the novel non-rinse conditioners did not improve the microtensile bond strength of new paste/paste RMGIC to dentin. The use of the novel non-rinse conditioners did enhance the bond strength of the traditional RMGIC to dentin.

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