BOND STRENGTH EVALUATION OF TWO RESIN CEMENTS WITH TWO ADHESIVES AND ANALYSIS OF MODE OF FAILURE

by

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INTRODUCTION

Resin luting agents have become the material of choice for luting most of the indirect restorations. All ceramic restorations tooth colored inlays, onlays, veneers, and crowns are now routinely bonded to the tooth using adhesive resin cements. These resin luting agents have the capacity to bond both to the tooth and to the restoration. This integration has been known to reduce microleakage at the restoration tooth interface, and also to lessen post-operative sensitivity, marginal staining, and recurrent caries.[1] Resin cements come in different modes of activation: auto/chemical cure, light cure, and the dual-cure modes.[2]

The bonding of indirect restoration with some resin luting agents requires the pretreatment of dentin with an adhesive system. Adhesive systems are manufactured as total-etch and self-etch systems. In keeping with dentists' time constraints and desires for easier-to-use products, manufacturers were stimulated to produce simplified products. The adhesive system initially comprised of the etchant, primer, and bonding agent in three separate bottles was reduced to two steps by combining the primer and bonding agent in one bottle with the etchant in a separate container.

Technique-sensitivity problems on the part of the operator and post-operative sensitivity complaints by the patient led manufacturers to develop self-etching systems. Self-etching systems work differently when compared with the total-etch systems. The total-etch systems remove the smear layer and de-mineralize the dentin, while the self-etch systems incorporate the smear layer while forming the resin dentin bond. Self-etch systems come as two-bottle systems, in which the etchant and the primer are combined in one bottle and the bonding agent separate, or as single-bottle/all-in-one systems where all the three steps are combined into one.

The two-step total-etch and the all-in-one adhesives have certain limitations when they are used in conjunction with chemical or dual-cured resins.[3-5] These issues include chemical incompatibility and hydrolytic degradation[6, 7] at the interface due to the permeability of these adhesives. The incompatibility is due to an acid- base reaction of the acidic monomers with amines, which are used in the initiator systems, such as the camphoroquinone/amine system in visible light curing adhesives or the amine/peroxide system in the chemical-cured adhesives. In both cases the concentration of the amine and the formed amine radical, which is responsible for the initiation of the polymerization, are decreased. The retardation of polymerization not only occurs in the adhesive layer but in the oxygen-inhibited surface zone that binds the composite to the adhesive. The acid-base reaction results in equilibrium between the protonized and the unprotonized forms of the amine and the acid. Therefore, the concentration of the amine has to be correctly adjusted to the concentration of the acid in the self-etch systems.[8] Adding to this, these single-bottle adhesives behave like semi-permeable membranes after polymerization.[8-11] There are water channels that originate from the surface of the hybrid layer and extend through the adhesive layer to reach the adhesive composite interface. These water channels have been given the term water trees by Tay and Pashley.[9] The osmotic gradient required for this type of reaction to occur has been blamed on the dissolved ions in the oxygen-inhibited layer of the polymerized adhesives.[10] The clinical ramifications are low-bond strength values and premature failure of the restoration.

Kerr Dental recently introduced a new resin luting agent, Nexus Third Generation (NX3), which utilizes a proprietary re-dox system claimed to eliminate the incompatibility problems noted with the previous generation of composite luting agent, Nexus Second Generation (NX2).

The aim of this study was to evaluate microtensile bond strengths and modes of failure of NX3 and NX2 with two different adhesive systems (two-step total-etch and one-step self-etch) after one week and after three months of storage. A light microscopic examination was conducted to examine the mode of failure.

The hypotheses of this study were: $H_0$1) There will be a difference in the microtensile bond strength between the two resin luting agents; H_A1) There will be no difference in microtensile bond strength between the two resin luting agents; $H_0$2) The type of adhesive used will influence the microtensile bond strength; H_A2) The type of adhesive used will not influence the microtensile bond strength; $H_0$3) The storage time will influence the microtensile bond strength; H_A3) The storage time will not influence the microtensile bond strength.

REVIEW OF LITERATURE

DENTIN BONDING

When Buonocore[11] in 1955 introduced enamel bonding, it was a major breakthrough in the world of adhesion. This provided an alternate means of achieving adhesion, when compared with the earlier methods of sacrificing tooth structure. When the same bonding technique was tried on dentin, it failed, because the dentin was an entirely different substrate when compared with enamel. Changes had to be made to compensate for the wetness of the dentin substrate.[12] Dentin etching with 37-percent phosphoric acid removes the smear layer and the mineral phase exposing the collagen fibrils, and this permeable layer facilitates the infiltration of the resin monomers into the collagen network. This resin-infiltrated zone is called the hybrid layer. This phenomenon was described by Nakabayashi et al. in 1982.[13] Dentin bonding became a huge success with the introduction of hydrophilic bonding agents.

Bonding to dentin involves the use of an acid, a primer, and a bonding agent. After achieving demineralization of dentin with the etchant, a primer, a bifunctional molecule having a hydrophobic and hydrophilic functionality, is applied. This is followed by the application of the hydrophobic bonding agent, which bonds to both the priming monomer and the restorative resin monomer.[14, 15] This has become known as the traditional, three-step total-etch bonding technique. With three-step total etch systems, the problem of maintaining the appropriate moistness of dentin was an issue.[16, 17] The weakest link in the resin dentin interface is the hybrid layer.[18, 19]

Tay et al., when comparing acid etched moist and dry dentin with water free primers, demonstrated that there was incomplete resin infiltration within the demineralized intertubular matrix. The result was a weak, collagen-rich zone that is susceptible to hydrolysis and microleakage.[20] This has led to problems of tooth sensitivity in clinical situations.[21] Technique sensitivity issues in relation to the moistness of dentin have also been known to occur with the etch and rinse systems. This problem also persisted in the two-step total etch adhesive systems that were developed to simplify the three-step bonding technique. To overcome this problem and also to reduce the dentists' time constraints, the self-etch systems were introduced.

The self-etching systems come as two types: two-step and one-step self-etch systems. The former has the etchant and primer combined in one bottle with a separate bonding agent, and the latter has the etchant, primer, and bonding agent all combined in one bottle.[22] Simplified adhesive systems are of two types: 1) etch and rinse single bottle systems, and (2) all-in-one self-etch adhesive systems. In the self-etching primers, the hydrophilic monomers used are also acidic to etch the dentin. However, the one-bottle etching systems typically use a more acidic primer ($pH<1$) than that of the two-step self-etching systems (pH 1.9-2.4) where etching and priming are combined into a single step. In addition, the one-step adhesives blend both hydrophilic and hydrophobic monomers with a relatively high concentration of solvent to keep them in solution. In this mixture, water is also essential as an ionization medium to enable self-etching activity to occur. When solvents evaporate from the surface of the adhesive, problems of monomer-solvent phase separation are seen, which create water droplets in the adhesive after polymerization.[23, 24] Hybrid layer formation is of crucial importance in maintaining the integrity of the resin-dentin bond. The main difference between the total etch and the self-etching systems is that the former removes the smear layer and demineralizes the dentin, whereas the latter incorporates the smear layer into the hybrid layer. The hybrid-layer thickness does not contribute to the bond strength. However, the quality of this layer is important, and how well the bonding resin impregnates the demineralized dentin is crucial.[25]

Many studies have been done to evaluate the microtensile bond strength of one-bottle adhesive systems comparing them with the total-etch three-step, total-etch two-step, and two-step self-etch systems. Results show that most of the one-bottle systems have lower bond strength when compared with the others.[26-29]

RESIN LUTING AGENTS

Indirect adhesive procedures constitute a large portion of contemporary oral rehabilitation procedures.[30] Metal and metal free inlays, crowns, veneers, orthodontic brackets, and even posts are now boned routinely using adhesive and resin luting agents.[31] Resin luting agents are capable of achieving a bond to the intaglio surface of restorations, with the use of adhesive systems that help them bond to dentin. Resin luting agents are a low-viscosity variant of composite restorative materials that contain a silanated filler and a resin such as BIS-GMA.[32] Their physical properties include low solubility in oral fluids, low potential for microleakage, and excellent tensile and compressive strengths.

Lambrechts and colleagues in 1991 classified composite luting agents according to viscosities and their initiating systems. This classification based on viscosities has slowly disappeared, and it is more appropriate to classify them based on initiating systems. Resin luting agents come in different modes of initiation, the auto/chemical polymerizing and the light and dual-cure initiating systems. The auto-polymerizing type is slowly fading from the market. Most of the resin luting agents available today are in the dual-cure initiating mode. There has been a growing concern regarding the incompatibility of chemical and dual-cure resin luting agents when they are used in conjunction with simplified adhesive systems.[5, 33]

INCOMPATIBILITY ISSUES

In order to understand the incompatibility of the adhesive systems with chemical or dual cured resin systems, it is important to understand the chemistry of the adhesive systems. The adhesive function of dental adhesives is twofold; they establish a bond to the enamel and dentin and secondly bind with the overlying composite. The latter has been shown to be one of co-polymerization. Regardless of whether the adhesive system is etch and rinse or a self-etch system, they all basically contain similar ingredients despite the number of bottles. Nevertheless, the proportional composition differs with the different classes of adhesive systems.[34]

Just as in composite filling and luting materials, an adhesive system traditionally consists of acrylic resin oligomers similar to those used in composite restorative materials. They also contain organic solvents, initiators, inhibitors, and some filler particles. The structure of a resin oligomer can be divided into three distinct parts: it has a polymerizable group, a spacer molecule, and a functional group. The polymerizable group will react with the other monomers of the adhesive and the restorative material by copolymerization and usually exhibit hydrophobic behavior. The functional groups usually exhibit hydrophilic properties. These monomers polymerize via a radical polymerization reaction. To initiate these reactions certain initiators are added; these can be benzoyl peroxide/tertiary amine in the chemical cured composites or photo-initiators like camphoroqinone/tertiary amines in the photopolymerization process. When both the above are used together, they comprise dual-cured systems. For a successful adhesive bond, not only should good bonding of adhesive to tooth occur, but a good copolymerization between the adhesive and the lining composite should be obtained. When the incompatibility issues are considered, copolymerization is affected, which may result in frequent failure of restorations.[34]

Simplified adhesive systems share a common characteristic in that they are somewhat acidic and have a hydrophilic layer, which is susceptible to hydrolytic degradation. During cementation of indirect restorations, the acidic groups from the oxygen-inhibited layer of the adhesive compete with the peroxides for the aromatic tertiary amines of the overlying resin. This results in an acid-base reaction between the adhesive and the resin luting agent.[35] The adverse interaction between single bottle adhesives and chemically cured composites has been well-documented by Tay et al.[36] The charge-transfer complexes that were formed between acidic monomers and the aromatic tertiary amines prevented the latter from participating in the redox reaction and impeded free-radical generation, which resulted in incomplete polymerization.

Light activation proceeds via the generation of free radicals from the activation of a photo-initiator, usually camphorquinone to its excited triplet stage. This is followed by the reduction of the activated photo-initiator by a less nucleophilic amine accelerator to form an intermediate excited complex, which releases free radicals on dissociation.[36] The chemical incompatibility between acidic resin monomers and chemical-cured composite was thought to be the result of a slower rate of polymerization and ideally should not occur when light-cure systems are used. However, incompatibilities with light-cure systems and simplified adhesives have also been reported. Regardless of the mode of polymerization, whether through light or chemical curing, there is a acid-base reaction that results in an equilibrium between the protonized and unprotonized form of the amine and the acid (Figures 1 and 2); therefore, the concentration of amine needs to be adjusted to the level of the acid present in self-etching systems.[8] Incompatibility exists even when the tertiary amines are substituted by other photo-accelerators. This has led researchers to believe that there may be other reasons for these problems to occur.

One of the reasons given by Tay[7, 36] was hydrolytic degradation. Hydrolysis is a chemical process that breaks covalent bonds within the polymers by the addition of water to ester bonds. Resin degradation is related to water sorption within the hybrid layer. Researchers have studied the water sorption of the simplified adhesive systems.[37-39] They have reported that hydrophilic resins had a higher water sorption than hydrophobic resins. Water sorption lowers the modulus of elasticity of the resins, which was thought to contribute to reductions in bond strength values. Combining hydrophilic acidic monomers into the bonded interface makes them more susceptible to hydrolytic degradation. Regardless of bonding techniques, water is drawn from the dentinal surface to the adhesive-resin interface. The name "water trees" was given by Tay[9] to describe this phenomenon. Tay demonstrated the effects of delayed activation on one- bottle, simplified adhesive systems, in which there was the presence of resinous globules formed along the fractured interface between the resin and dentin. Water movement across the cured adhesive layer may occur in the regions of increased concentrations of dissolved inorganic ions; uncured, water soluble, hydrophilic resin monomers; and dissolved collagen/proteoglycan components within the air-inhibited layer of the adhesive. These water-soluble agents may lower the local water concentrations and thereby establish an osmotic gradient causing water to move from a region of low solute concentration (the dentinal tubules) to a region of higher solute concentration (the air inhibited layer of the adhesive uncured composite interface). This may also give rise to osmotic blistering. As this blistering was evident in the chemical-cured composites, the water permeation may be partially responsible for the ineffectiveness of simplified adhesives and chemical-cured resins.

TESTING METHODOLOGY

Although clinical trials are the ultimate test for any dental material, sometimes it is difficult to differentiate the reasons for failure due to the simultaneous impact of diverse variables on restorations within the oral cavity. Laboratory testing can provide a narrow means of evaluation. The option is available to test one variable and keep some others constant,[40] although a direct correlation to the clinical situation is not possible. Laboratory testing does provide a faster and more convenient alternative for screening dental materials, and bond-strength tests are often used to test adhesive systems. The rationale behind this methodology is that the stronger the adhesion between the tooth and biomaterial, the better it will resist stress imposed by resin polymerization and oral function . Many different types of bond strength tests exist, but the shear and microtensile are the most commonly reported.

Some of the advantages of the microtensile testing are that:[41]

- Testing of a very small area is permitted.
- Regional bonding strengths can be measured.
- Higher interfacial bond strengths can be measured.
- Irregular surfaces can be tested.
- There are more adhesive failures and fewer cohesive failures.
- Means and variance can be calculated for a single tooth.
- Many specimens can be obtained from fewer teeth.

Some disadvantages of the microtensile bond strength testing methodology are:

- The methodology is labor intensive.
- Small samples can dehydrate rapidly.
- It is technically demanding.
- It uses special equipment.

One area of concern regarding the microtensile strength testing is the trimming of the slabs into hour-glass shaped specimens.[42] In this procedure, a concern developed that added stress would weaken the bonded interface and result in skewed values for tensile strength. Thus, a non-trimming method was introduced in which each slab was cut with the top half made of composite and the bottom half of dentin. Utilizing this technique, a single molar tooth may yield 20 to 25 specimens with cross-sectional areas of 0.7 mm^2 to 1.2 mm^2 depending on the size of the tooth.[42] After the tooth has been bonded and a resin composite core has been built up, the tooth can be sectioned vertically five or six times.

The tooth is then rotated 90º and another four or five sections can be made, which will result in 20 to 25 beams that remain attached to the base. Each beam is labeled prior to separation, which facilitates statistical evaluations. This technique of regional measurement of resin bonded to tooth substrate is known as an array.[43] Eckert and Platt[44] investigated the need to account for correlations between beams to avoid overstating statistical significance of study results. In this study, only the four beams from the central portion of the tooth were marked and used for microtensile testing. A random effect was included to account for the correlations among beams from the same tooth. Following the microtensile strength test, all beams were analyzed under the stereomicroscope to determine the mode of failure.[1]

MATERIALS AND METHODS

Sixty-four non-carious, non-restored human molar teeth were collected under an IUPUI/Clarion IRB approved protocol. The occlusal faces of the crowns were sectioned to expose dentin using a low-speed speed saw with a diamond blade. The dentin surface was ground with a 340-grit SiC paper under deionized water flow. The absence of enamel was verified using a stereomicroscope (Nikon Measurescope UM-2, Nikon Inc., Melville, NY). The 64 teeth were then divided into four groups of 16 teeth. Two adhesives, the total-etch Optibond Solo Plus (SDS Kerr Corp., Orange, CA) (Figure 3) and the self-etch Optibond All In One (AIO) (Figure 4) (SDS Kerr), and two cements, Nexus Second generation (NX2) (Figure 5), and Nexus Third generation (NX3) (Figure 6) (SDS Kerr), were then evaluated (Table I). The four groups were then restored following manufacturers' instructions in the following manner.

COMPOSITE DISC PREPARATION

A Teflon mold of 2-mm thickness was taken and placed over a glass plate; the mold was then filled with composite resin (Premise A2, SDS Kerr) (Figure 7). A Mylar strip was taken and placed over the composite resin. A second glass plate was taken and pressed over the Mylar strip firmly. Then, the glass plate was removed and the composite resin cured using an LED Demetron 1 unit (SDS Kerr) with an intensity $870 mW/cm^2$ for 40 seconds.

RESTORATION OF TEETH

Nexus 3 with Optibond Solo Plus

For this group of 16 teeth, a 37-percent phosphoric acid gel etchant was used. The teeth were acid etched for 15 seconds, washed for 15 seconds, then blot-dried using absorbent paper. After acid etching, this group was treated with Optibond Solo Plus (SDS Kerr). The adhesive was applied with a microbrush for 15 seconds with a light brushing motion and air thinned for 3 seconds using canned air (Dust Off, Falcon Safety Products, Sommerville, NJ) to achieve a visibly uniform layer. The surface was then light-cured for 20 seconds. Two composite resin discs of 2 mm each were then luted to the treated dentin surface with NX3 resin cement with a load of 640 g. The resin was activated using an LED Demetron 1 unit (SDS Kerr) 870mW/cm^2 from all four sides and also from the top. The restored specimens were then stored in distilled water at 37° C until the beams were obtained.

Nexus 2 with Optibond Solo Plus

This group was treated as described for NX3 with Optibond Solo Plus (SDS Kerr), except the NX3 was replaced by NX2.

Nexus 3 with Optibond All In One

This group of 16 teeth was taken and blot dried, following which Optibond All In One (SDS Kerr) adhesive was applied using a microbrush for 15 seconds with a light brushing motion and air thinned for 3 seconds using canned air (Falcon) to achieve a visibly uniform layer. The surface was then light-cured for 20 seconds. Two composite resin discs of 2 mm each were then luted to the treated dentin surface with NX3 resin cement with a load of 640 g. The resin was activated using an LED Demetron 1 unit (SDS Kerr) 870mW/cm^2 from all four sides and also from the top. The restored specimens were then stored in distilled water at 37° C until the beams were obtained.

Nexus 2 with Optibond All In One

This group was treated as described for NX3 with All In One (SDS Kerr) except the NX3 was replaced by NX2.

BEAM PREPARATION

The same procedure was followed for each of the four groups.

The 16 teeth for each group were taken and 64 beams (0.8x0.8x7±1mm) were obtained for each adhesive/resin cement combination using a non-trimming technique with a low-speed saw. Each tooth was sectioned multiple times in two planes at 90° to each other (Figures 8, 9 and 10). Four beams for each tooth were obtained, two beams for the immediate and two for the three months measurement. The beams were then stored in distilled water until testing. The water for the three month measurement was changed every week to prevent bacterial growth (Figure 11).

MICROTENSILE TESTING

An individual beam was placed on the notched jig for microtensile testing using cyanoacrylate glue (Zapit, Dental Venture of North America, Corona, CA) and was subjected to a tensile force in a universal testing machine (Instron, Norwood, MA) (Figure 12) at a crosshead speed of 1 mm/min using a 125N load cell. The bond strength

(MPa) was determined by dividing the maximum load to failure (N) and the cross sectional area (mm^2).

ANALYSIS OF MODE OF FAILURE

Each specimen was then analyzed for mode of failure using a Nikon Measurescope UM-2 and failure mode was classified as follows:

Cohesive Failure in Resin

There was no dentin visible. This failure mode included a cohesive failure of the adhesive, cohesive failure of the luting agent, or a failure between the adhesive and the luting agent.

Adhesive Failure

There was evidence of only dentin on the surface. This mode included a failure between the dentin and the adhesive.

Mixed Failure

A failure was classified as mixed when there was evidence of dentin and also there was some evidence of resin on the surface, so that two failure modes were evident, one in the interface between dentin and adhesive, and the other a cohesive failure in resin or luting agent.

A category for cohesive failure in dentin was not used because this failure mode was not seen.

SEM ANALYSIS

The dentin portions of the beams to be analyzed were glued on to the stubs using cyanoacrylate glue, and the beams were spattered with gold using a Denton vacuum with a gold target and examined under the scanning electron microscope (JEOL 5310 LV, Tokyo, Japan).

STATISTICAL METHODS

As some beams debonded prematurely, comparisons between the treatment combinations for differences in microtensile bond strength were performed using a Weibull-distribution survival analysis, using the force required for bond failure in place of the usual time-to-event seen in typical survival analyses. Beams that debonded before placement on the testing machine were accommodated in the survival analysis model as left-censored observations. The survival analysis model included a random effect to account for the correlations among beams from the same specimen, and such survival analysis models are often called frailty models.[45] Descriptive analysis was done and means were obtained for all groups.

Comparisons between the treatment combinations for differences in the failure mode were performed using Fisher's Exact tests. Most beams from the same tooth had the same failure mode; only one observation from each tooth was included in the analysis. If the two beams had different failure modes, the mixed mode was used in the analysis. Beams that debonded prematurely are not included in the failure mode analysis.

SAMPLE SIZE JUSTIFICATION

Using data from a previous study,[44] the correlation among beams from the same specimen was estimated to be 0.3. The standard deviation estimates were 8 MPa using immediate debonding (pilot study) and 15 MPa after three-month-storage. With a sample size of 16 teeth [46] per cement-adhesive combination, with each tooth divided into four beams, where two beams from each tooth were to be tested immediately and two beams to be tested after three months (32 beams per cement-adhesive-time combination), the study had an 80 percent power to detect a difference in microtensile bond strength of 6.7 MPa between any two cement-adhesive combinations for immediate testing, a difference of 12.4 MPa between any two cement-adhesive combinations for testing after 3 months, and a difference of 8.4 MPa between the two times for any cement-adhesive combination, assuming a 5-percent significance level for each test.

RESULTS

MICROTENSILE BOND STRENGTH RESULTS

For the microtensile bond strength summary statistics, beams that debonded prematurely were given a value of 0.5 (Table II).

Standard Error

Because of the correlations between beams, the standard errors are from an ANOVA with a random effect to properly account for the correlations between beams.

Weibull Parameters

The Weibull location parameters are compared to evaluate differences in survival time. The Weibull scale parameters in this study are allowed to differ between groups so that the fitted survival curves are allowed to cross. Due to the complexity of interpreting the shape and location parameters, the Weibull moduli and characteristic strength were determined. The Weibull modulus is a measure of variation of strength, and the characteristic strength is the strength when the survival strength falls below 40 percent (Table III).

Level of Significance for Groups

NX3 Solo Plus immediate had significantly higher bond strength than the following groups: NX3 Solo Plus three months; NX3 All In One immediate; NX3 All In One three months; NX2 All In One three months; NX2 Solo Plus immediate, and NX2 Solo Plus three months.

NX2 All In One immediate had a significantly higher bond strength than the following: NX3 All In One immediate; NX3 All In One three months; NX2 All In One three months; NX2 Solo Plus immediate, and NX2 Solo Plus three months.

NX3 Solo Plus three months had significantly higher bond strength than the following: NX3 All In One three months; NX2 All In One three months; NX2 Solo Plus immediate, and NX2 Solo Plus three months

NX3 All In One immediate, NX3 All In One three months, and NX2 All In One three months had a significantly higher bond strength than NX2 Solo Plus three months (Table IV).

Kaplan Meier Survival Plot

This was also performed to determine the survival probability (Figure 14).

MODE OF FAILURE RESULTS

NX2 Solo Plus three months and NX2 Solo Plus immediate had a significantly higher percentage of teeth with mixed failure than all other groups. No other groups had a significantly different failure mode (Table V).

SEM EXAMINATION

Random specimens from each group were examined under the scanning electron microscope to characterize the mode of failure (Figure 15 to Figure 30).

FIGURES AND TABLES

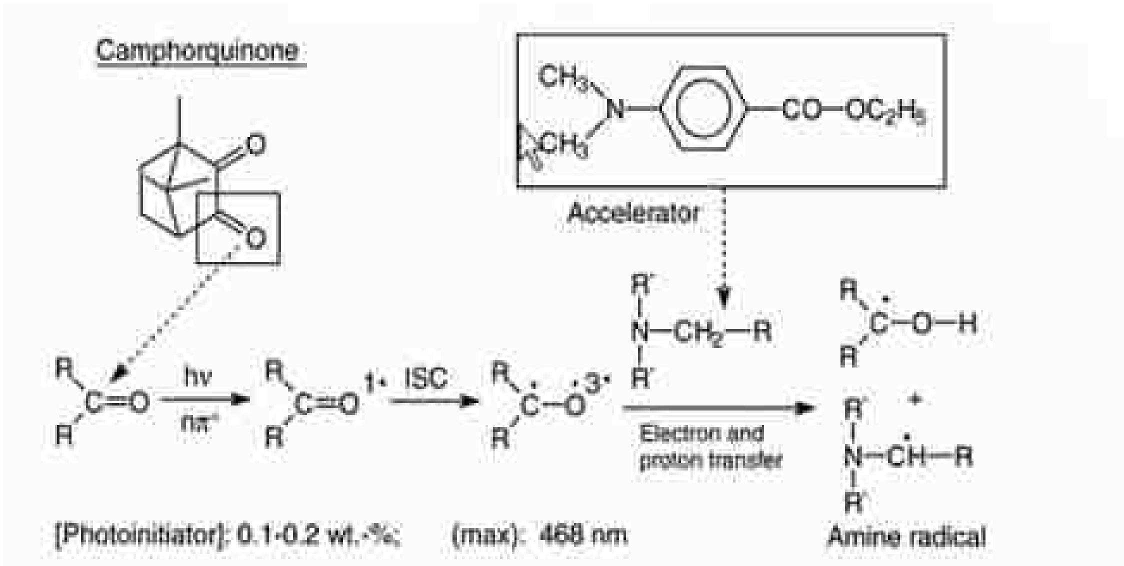

FIGURE 1a. The normal photopolymerization process generating an amine radical.

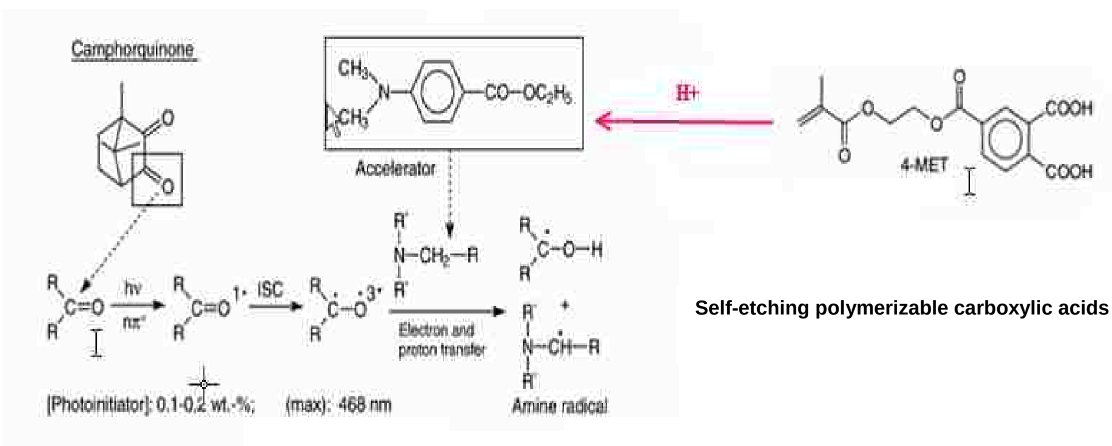

FIGURE 1b. Diagram showing hydrogen transfer occurs from the polymerizable acid groups of the monomers of the adhesive system to the tertiary amines preventing deprotonation of the amine to generate an amine radical in a photopolymerization reaction.

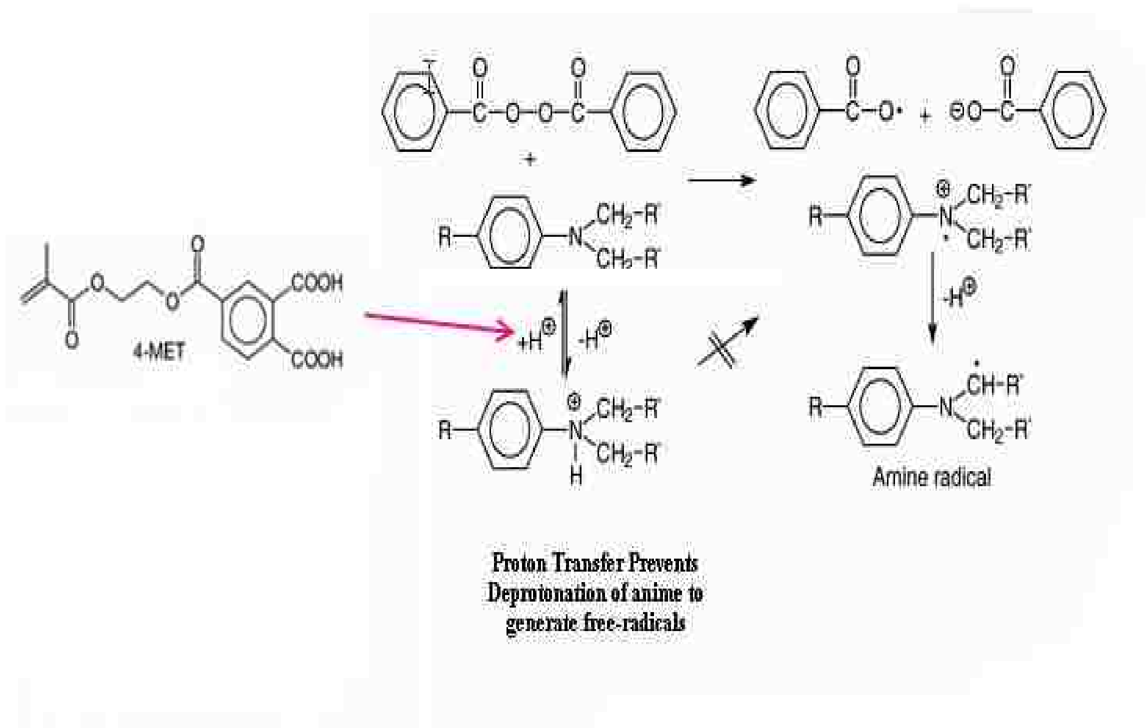

FIGURE 2. Diagram showing the hydrogen transfer from polymerizable acid groups of the monomers of the adhesive system to the tertiary amines preventing deprotonation of the amine to generate the amine radical in the chemical polymerization reaction.

FIGURE 3. Optibond Solo Plus, two-step, total-etch adhesive system, with phosphoric acid gel etchant.

FIGURE 4. Optibond All In One, single-bottle self-etch system.

FIGURE 5. NX2 resin luting agent.

FIGURE 6. NX3 resin luting agent.

FIGURE 7. Premise composite resin used to restore teeth. Arrow showing the 2-mm composite disc prepared with this composite resin.

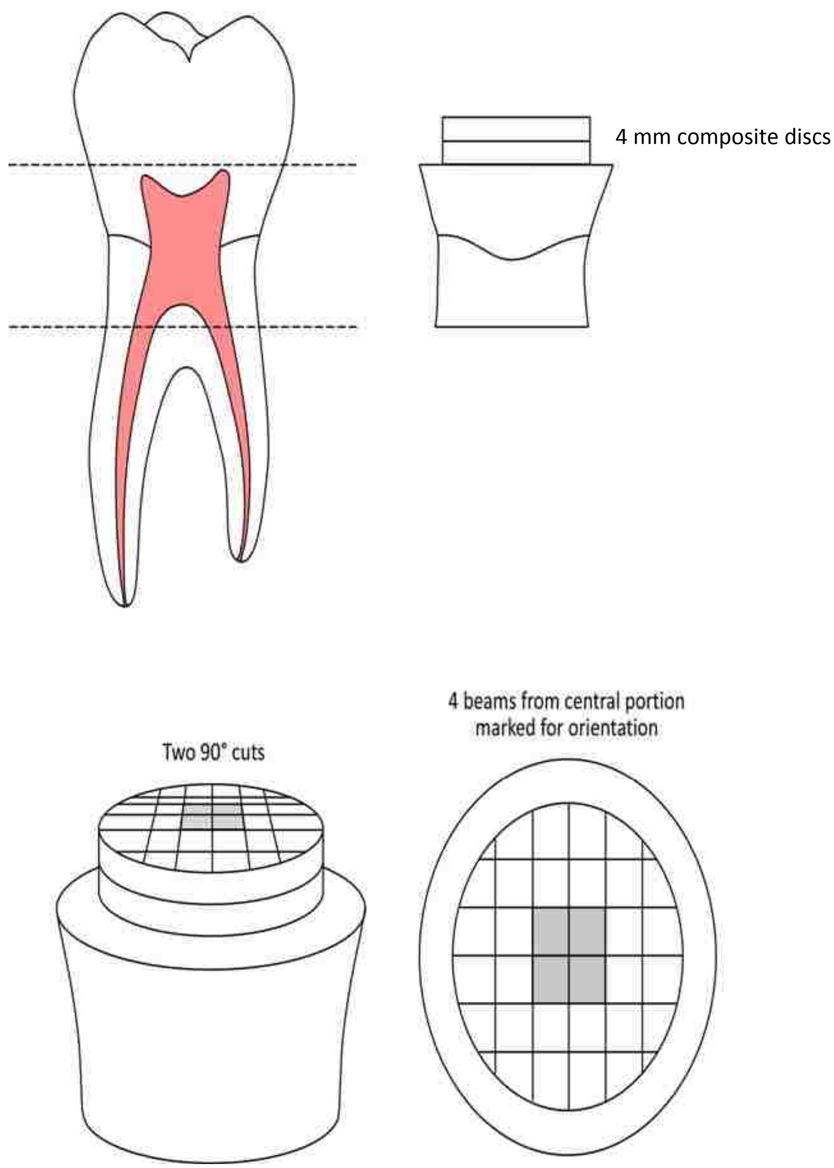

FIGURE 8. Schematic diagram of beam preparation.

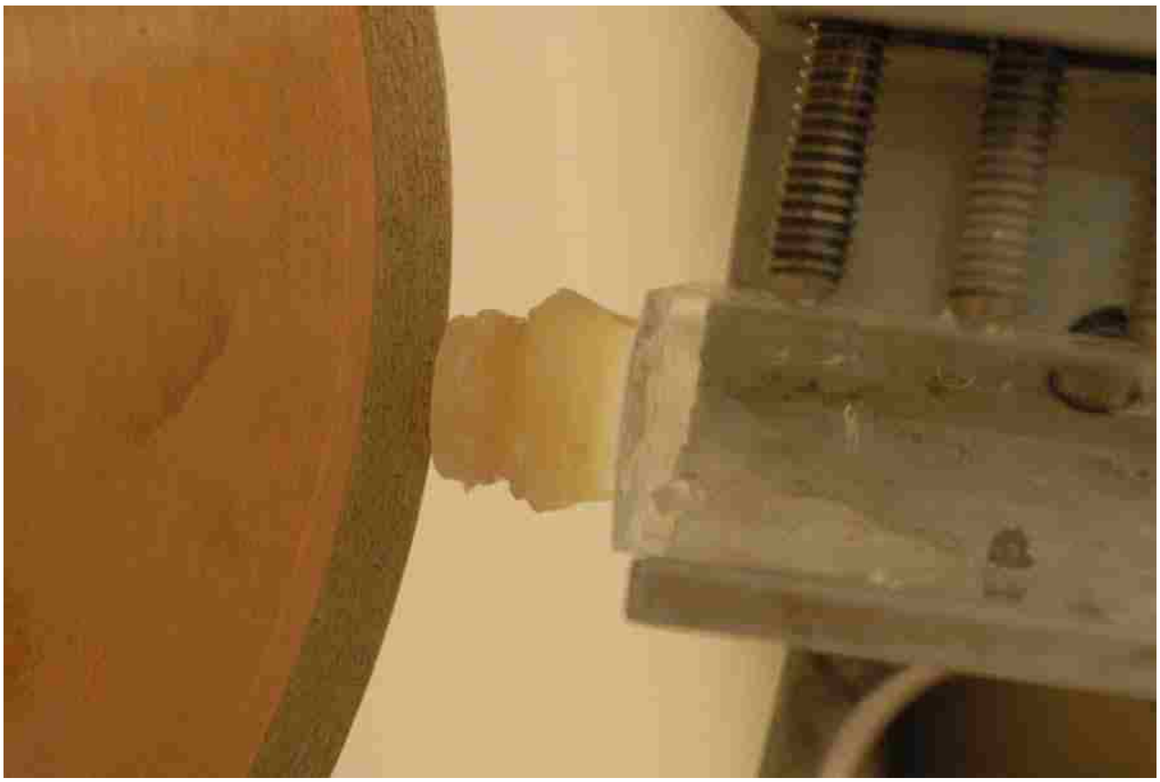

FIGURE 9. Beam preparation.

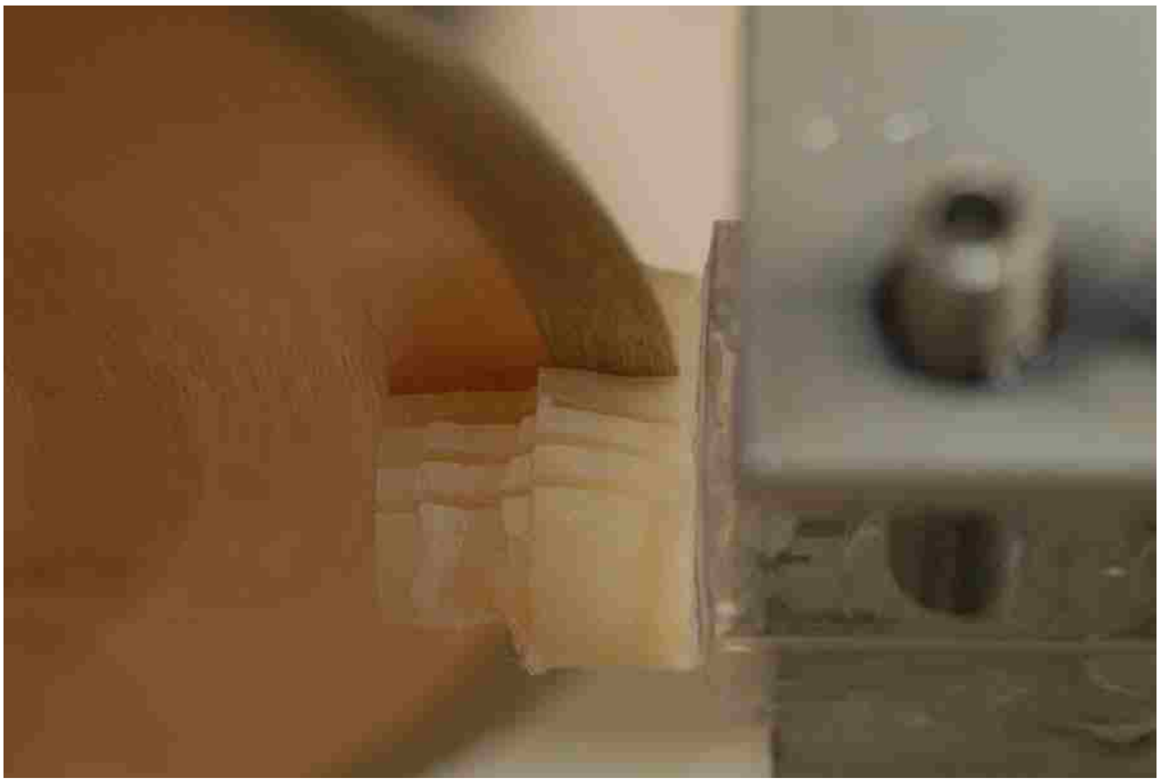

FIGURE 10.   Beam preparation.

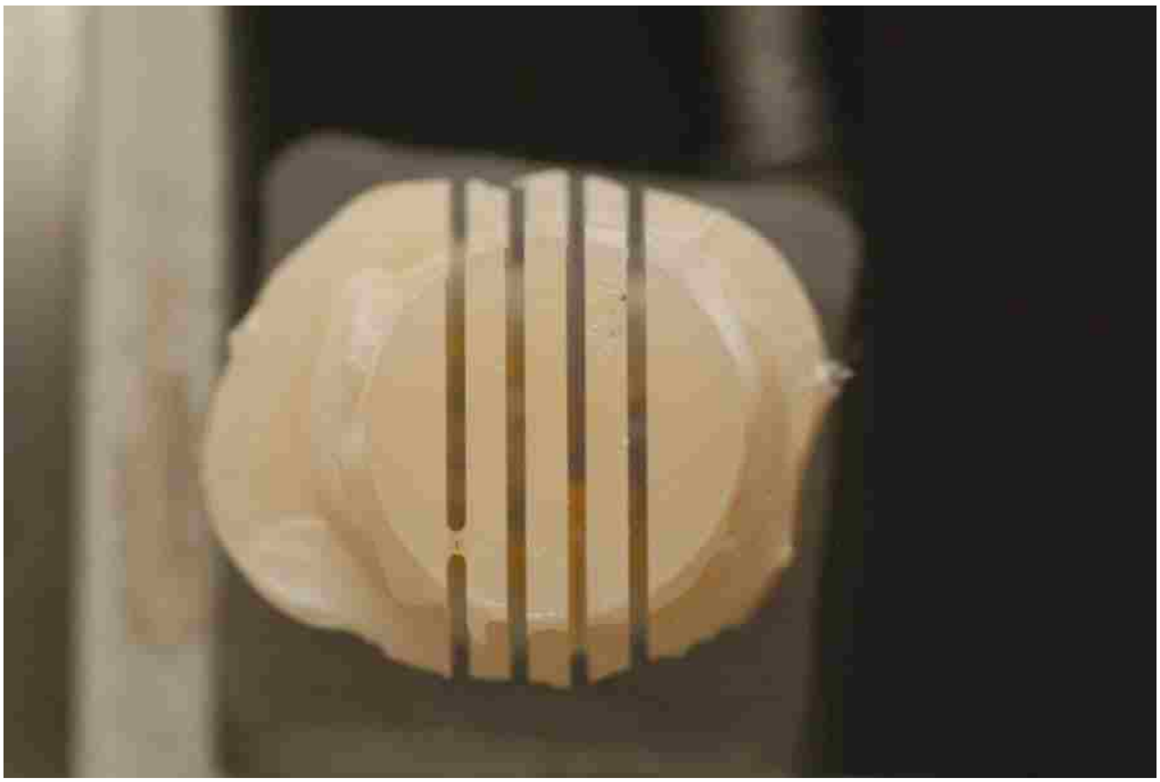

FIGURE 11. Beam preparation.

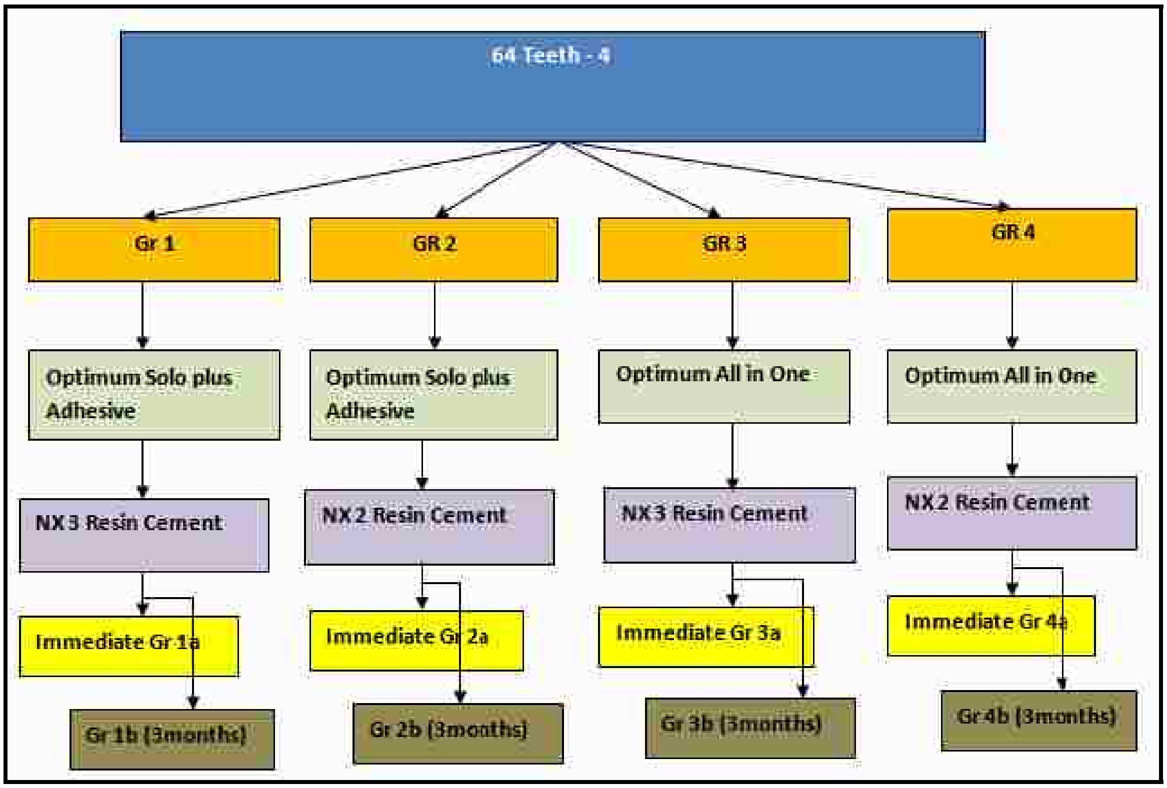

FIGURE 12. Division of groups after beam preparation.

FIGURE 13. Beam mounted on the universal testing machine.

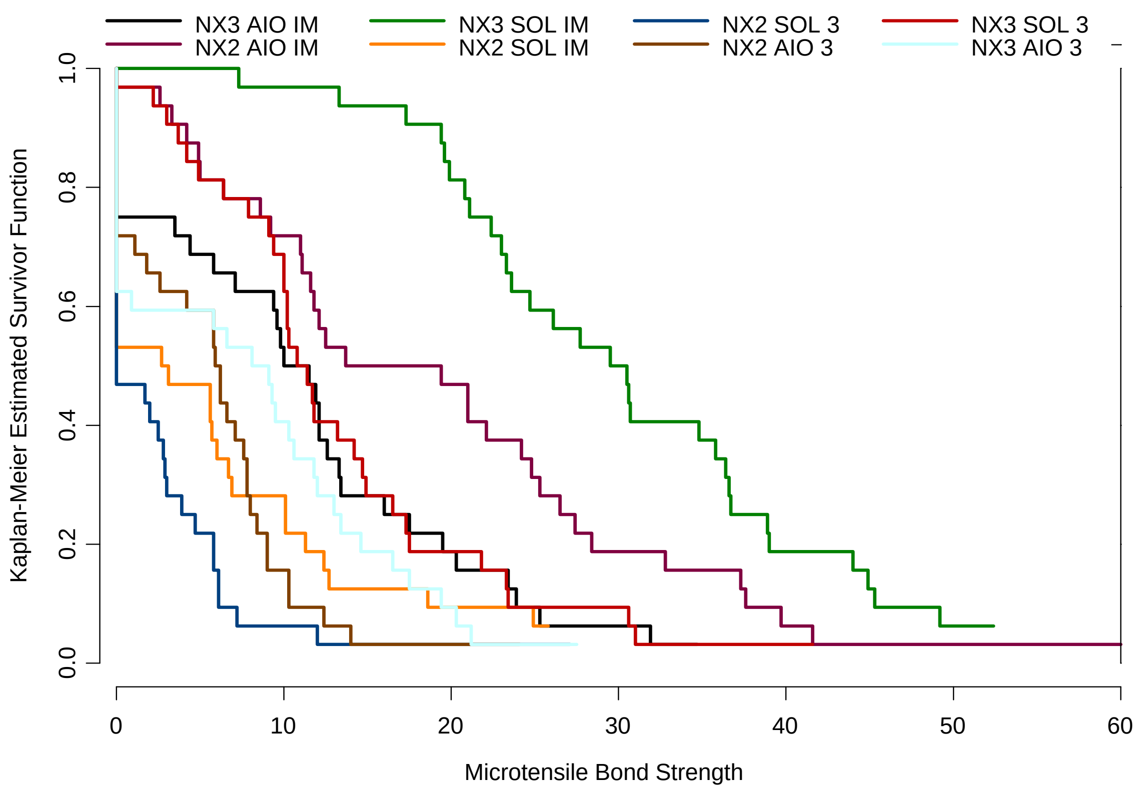

FIGURE 14. Kaplan-Meier survival plot.

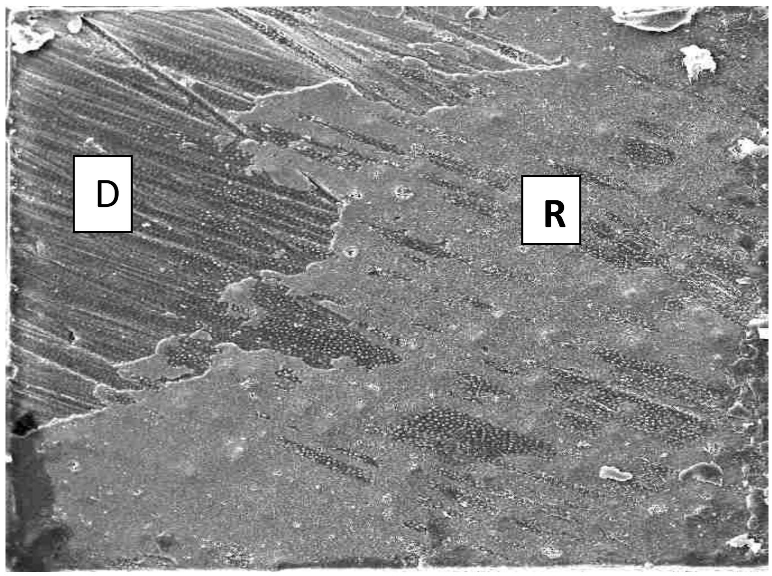

FIGURE 15. SEM image of NX3 Solo Plus immediate specimen at X100. The image shows a mixed failure with dentin (D) and resin (R).

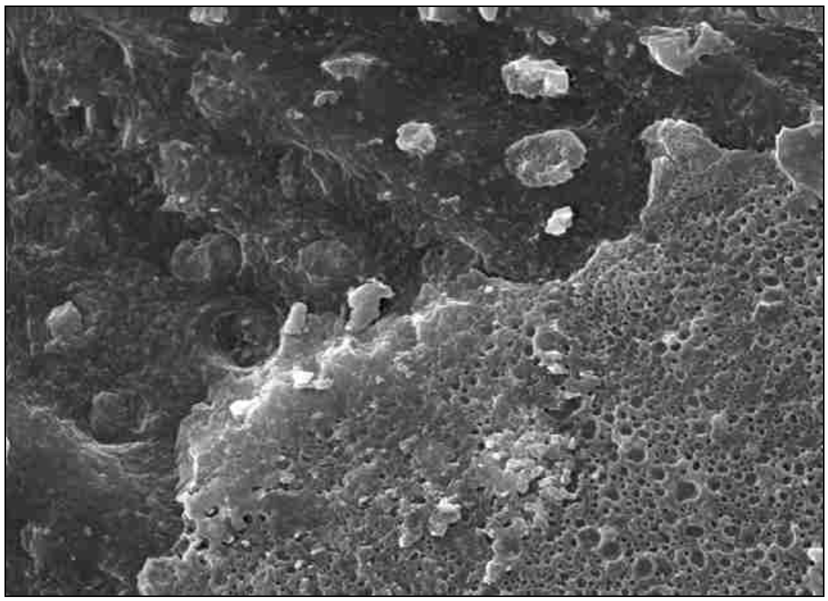

FIGURE 16. SEM image of NX3 Solo Plus immediate specimen at X3500 showing resin, and area of dentinal tubules.

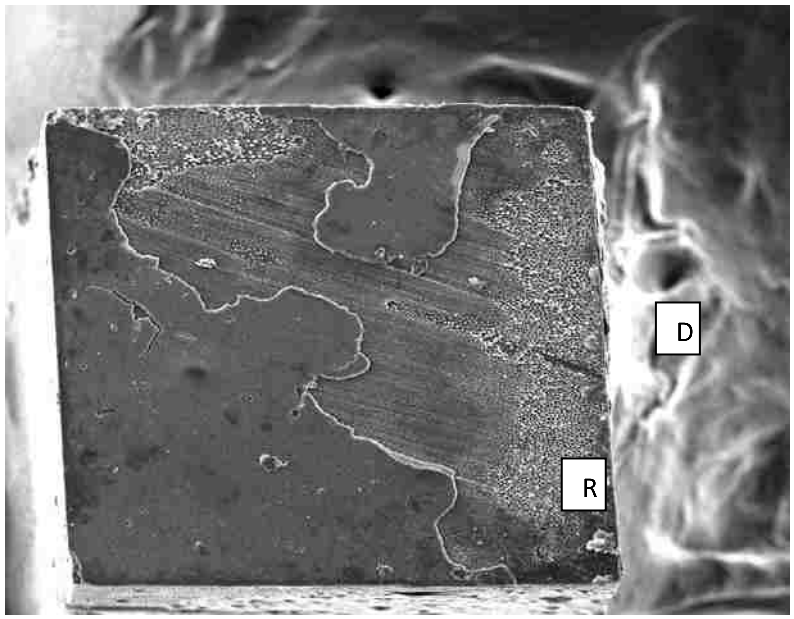

FIGURE 17. SEM image of NX3 Solo Plus three-month specimen at X100. Mixed failure showing dentin along with resin.

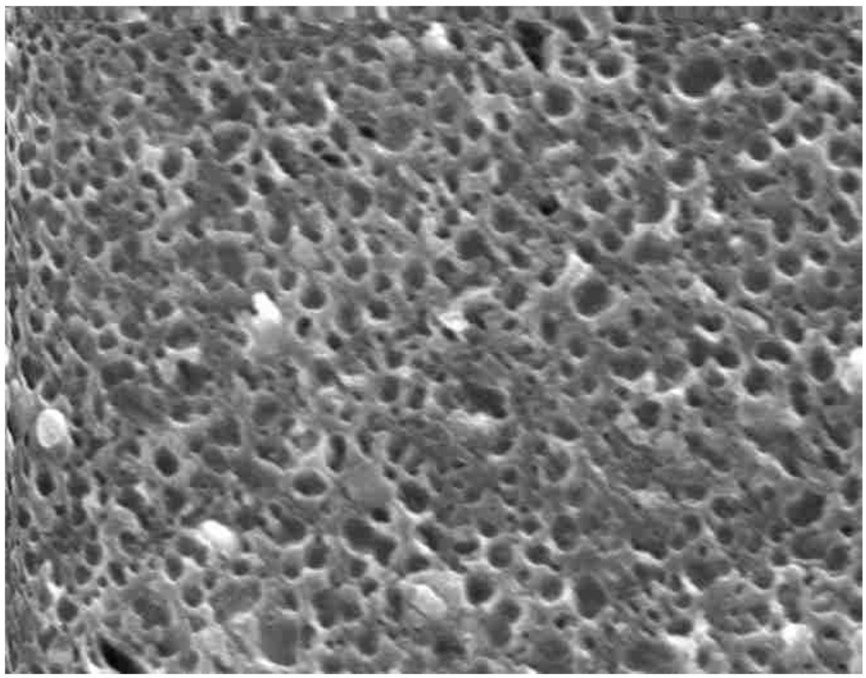

FIGURE 18. SEM image of NX3 Solo Plus three-month specimen at X1000. Here are signs of water blisters, giving the honeycomb appearance.

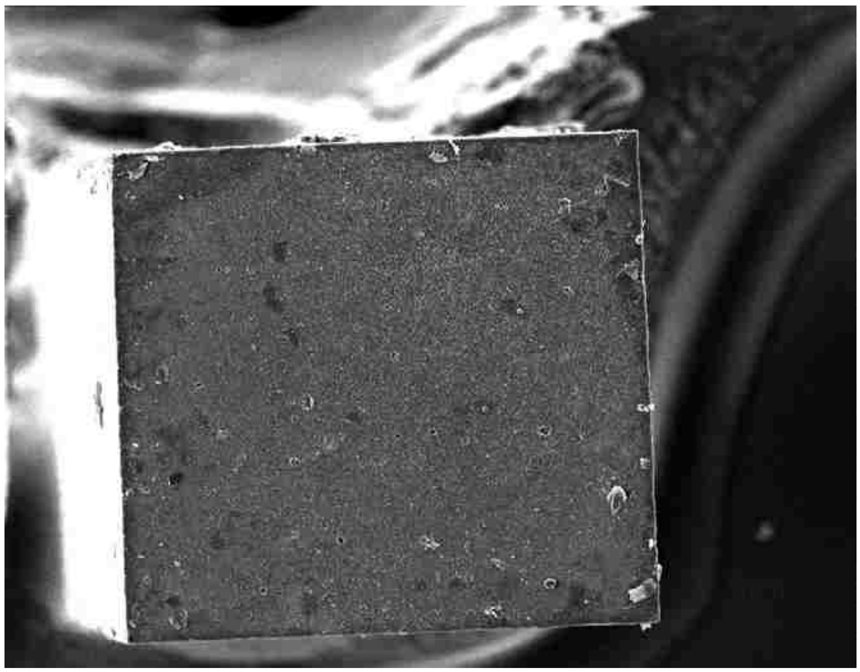

FIGURE 19. SEM image of NX3 All In One immediate specimen at X100 showing cohesive failure in resin.

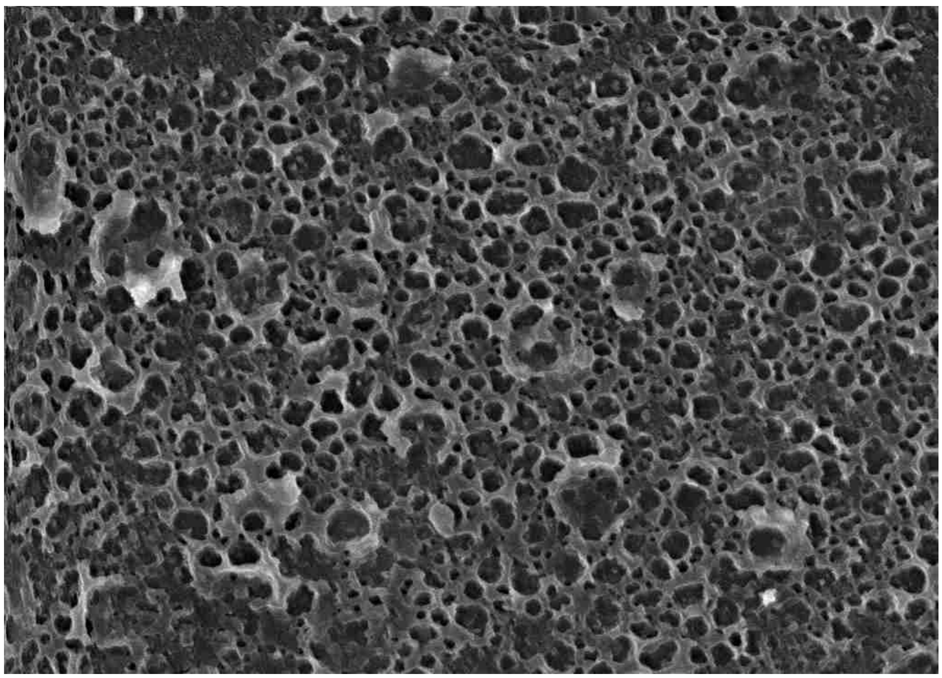

FIGURE 20. SEM image of NX3 All In One immediate specimen at X5000. Shown here, evidence of water blisters giving the typical honey-comb appearance.

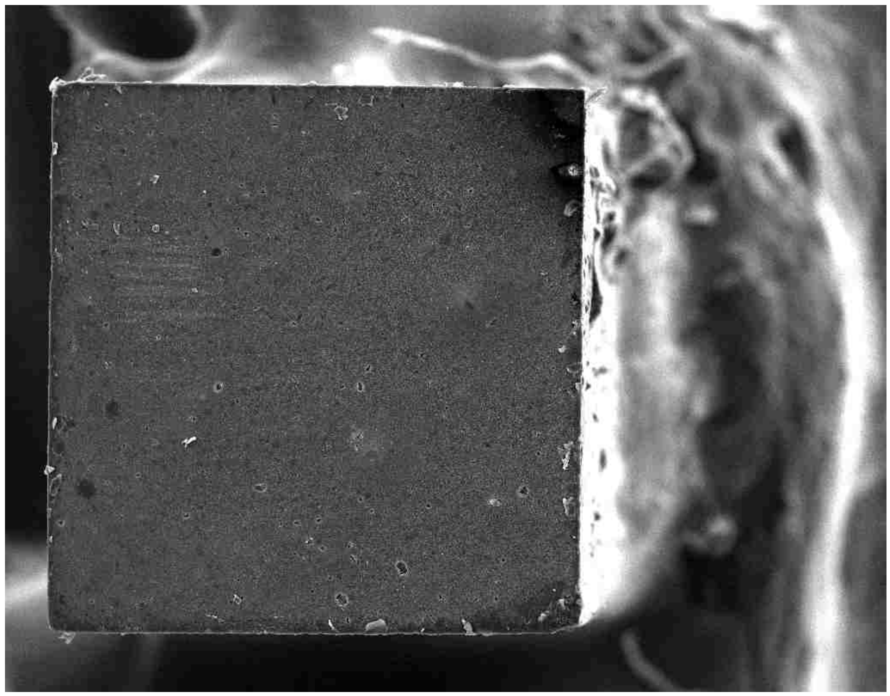

FIGURE 21. SEM image of NX3 All In One three-month specimen at X100 showing cohesive failure in resin.

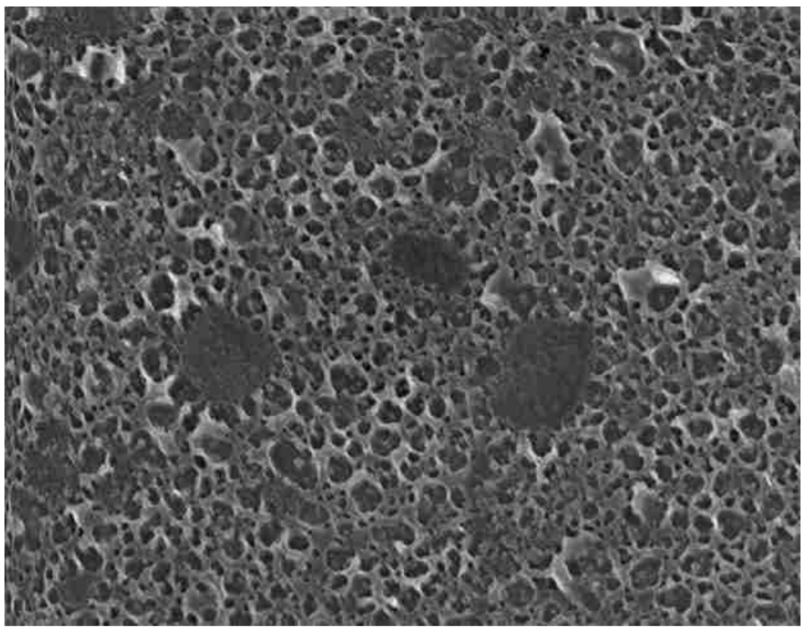

FIGURE 22. SEM image of NX3 All In One three-month specimen at X5000 showing osmotic blistering.

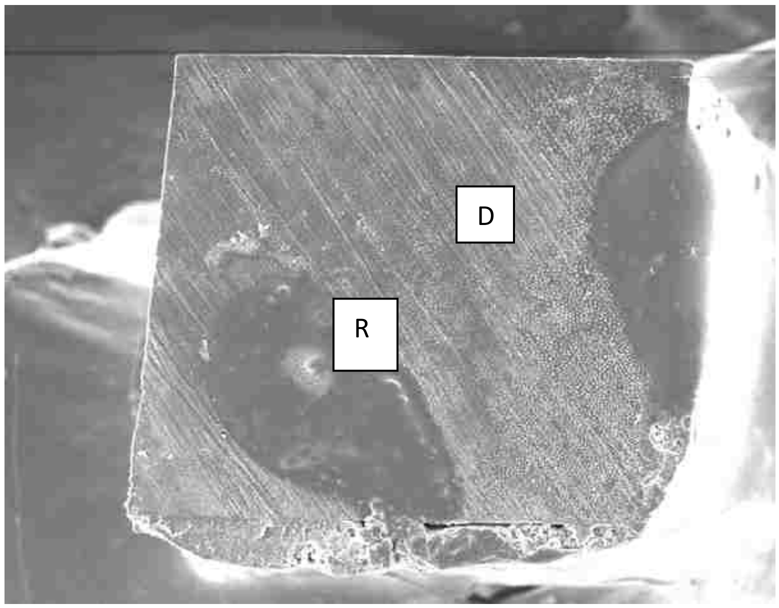

FIGURE 23. SEM image of NX2 Solo Plus immediate specimen at X100 showing mixed failure.

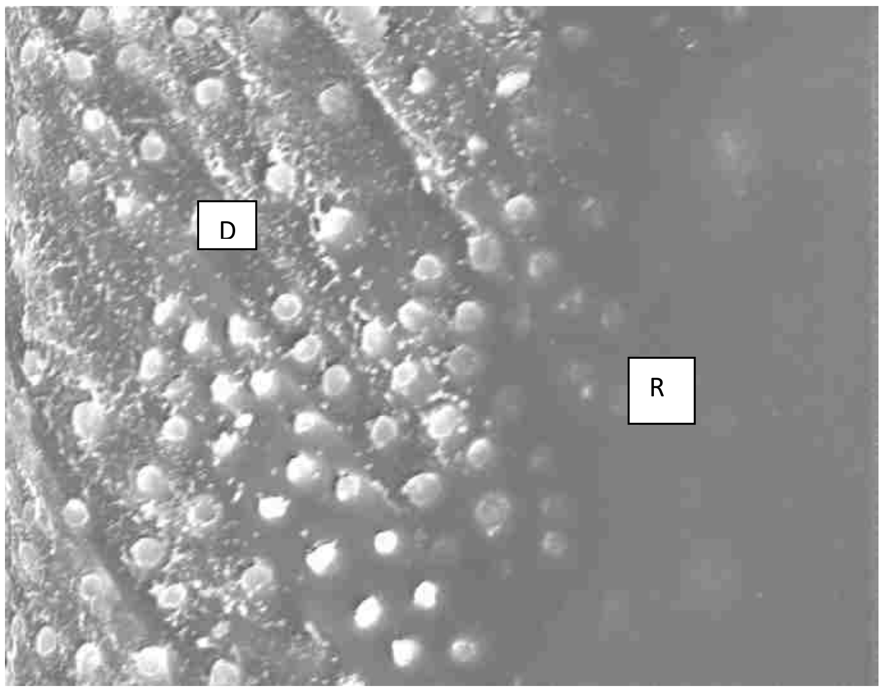

FIGURE 24. SEM image of NX2 Solo Plus immediate specimen at X1500 showing resin and resin plugged dentinal tubules.

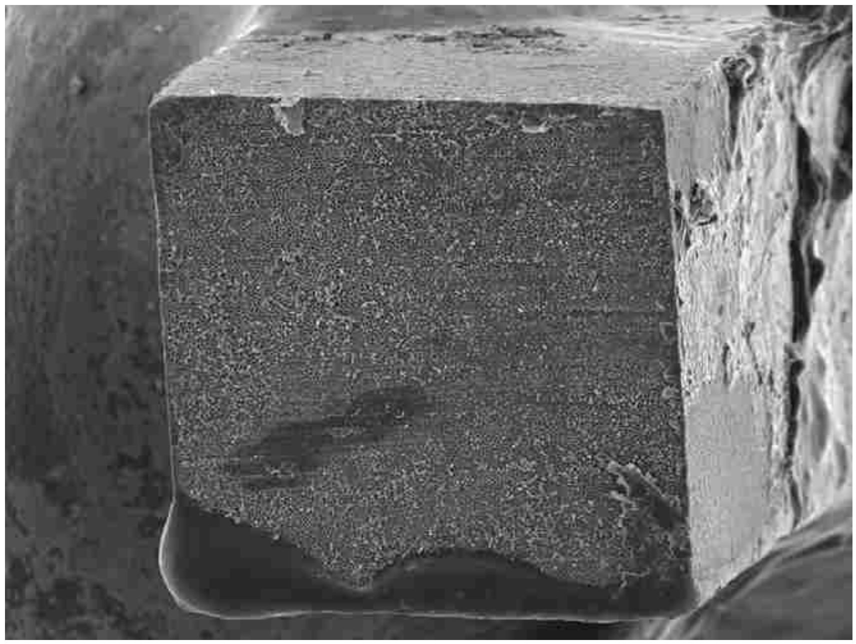

FIGURE 25. SEM image of NX2 Solo Plus three-month specimen at X100 showing mixed failure.

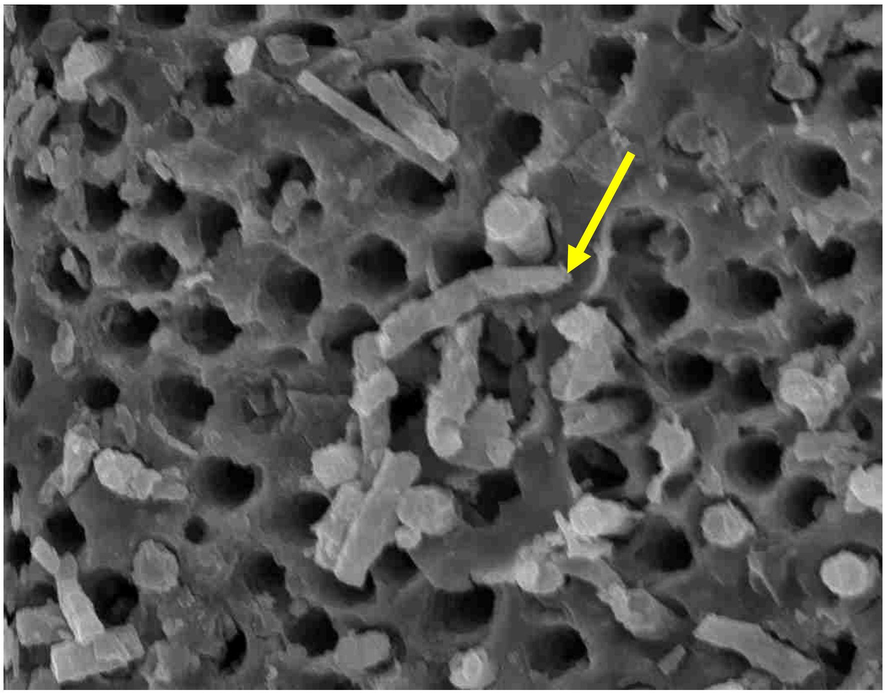

FIGURE 26. SEM image of NX2 Solo Plus three-month specimen at X2000 showing dentinal tubules with long resin tags pulled out of the tubules.

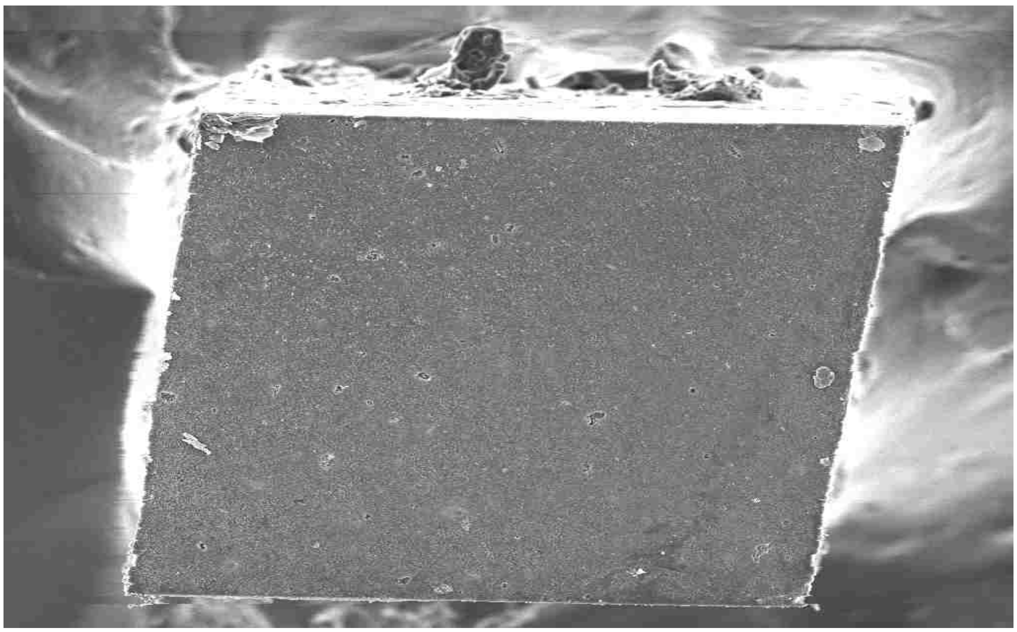

FIGURE 27. SEM image of NX2 All In One immediate specimen at X100 showing cohesive failure in resin.

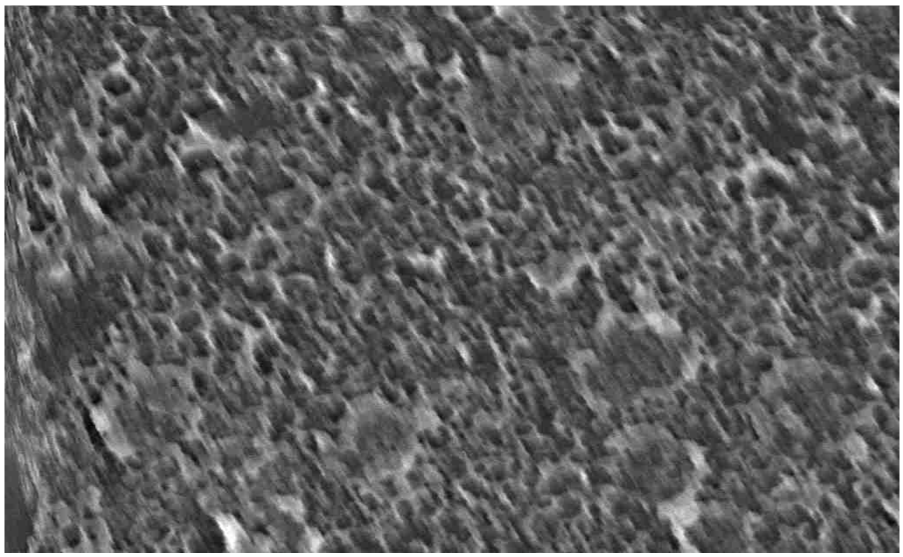

FIGURE 28. SEM image of NX2 All In One immediate specimen at X5000 showing cohesive failure in resin.

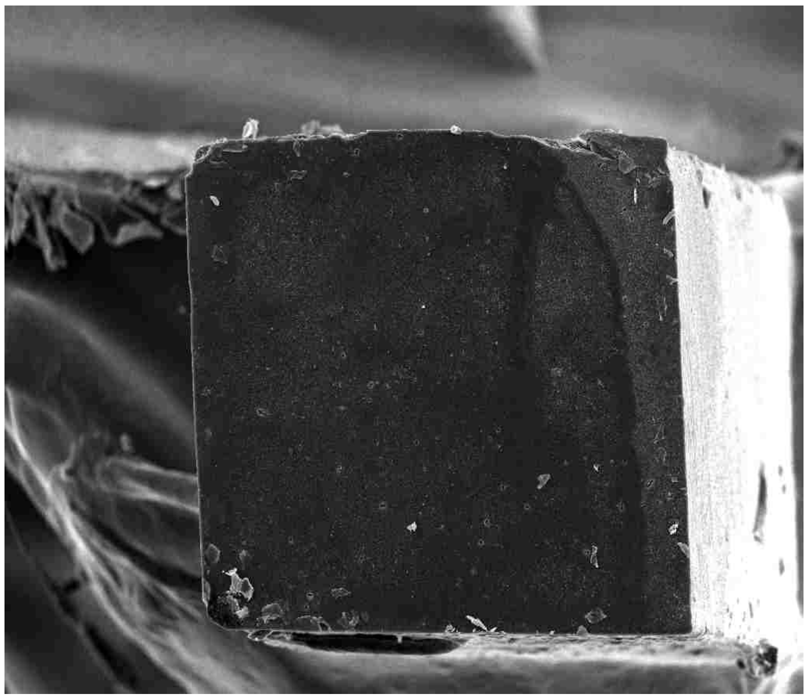

FIGURE 29. SEM image of NX2 All In One specimen after three months at X100 showing cohesive failure in resin.

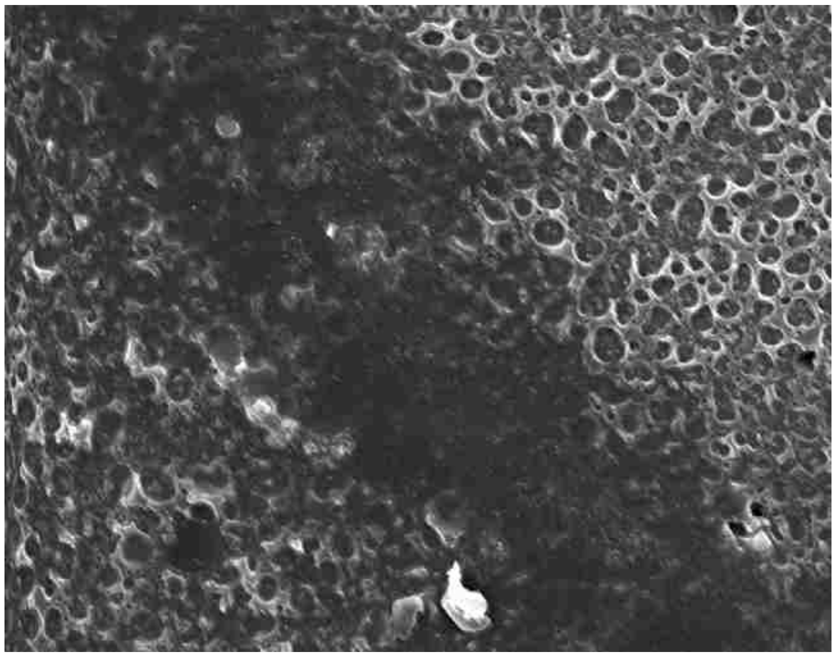

FIGURE 30. SEM image of NX2 All In One specimen after three months at X3500 showing the honeycomb appearance of the resin globules.

TABLE I

Materials used to restore the teeth

Material	**Manufacturer**	**Composition**	**Lot numbers**
NEXUS 2 Base Clear Catalyst Clear	KERR	Monomers of methacrylic Acid esters, Ba-Al-borosilicate, chemical and photo initiators	#2878837 #2875422
NEXUS 3	KERR	Composition not available	#2944881 #2945276
PHOSPHORIC ACID GEL ETCHANT		37.5% Free phosphoric acid	#2954525
OPTIBOND SOLO PLUS	KERR	37.5% H_3PO_4 Bis –GMA,GPDM,GDM,HEMA, Ethenol,water,filler,CQ	#448447
OPTIBOND ALL IN ONE	KERR	Hexafluoroglutaric anhydride-glycerodimethacrylate adduct, glycerol phosphate dimethacrylate, ethanol, water, 2(ethylhexyl)-4-(dimethylamino) dimethacrylate, butylhydroxytoluene, filler (fumed SiO_2, barium aluminoborosilicate Na_2siF_6	#2721702
PREMISE COMPOSITE RESIN	KERR	69 vol% of 30-50µm pre polymerized filler(PPF), 0.4µm barium glass, 0.02µm silica nano particles, ethoxylated bis-openol A-dimethacrylate, TEGDMA.	#457851 #444853 #2774442 #2878940

TABLE II

Means of all groups

Group	# teeth	# beams	% early debond	Mean	SE	Min	Max	Weibull parameters	
								Location	Scale
NX2 AIO IM (Nexus 2 All In One immediate)	16	32	3	19.3	3.3	0.5	60.0	2.80	0.273
NX2 AIO 3MON(Nexus 2 All In One 3-month group)		32	28	5.8	1.2	0.5	24.1	1.23	0.286
NX2 SOL IM (Nexus 2 Solo Plus immediate)	16	32	47	6.3	1.5	0.5	25.8	0.88	0.823
NX2 SOL 3MON(Nexus 2 Solo Plus 3-month group)		32	53	3.2	1.2	0.5	27.1	0.27	0.537
NX3 AIO IM (Nexus 3 All In One immediate)	16	32	25	11.3	2.2	0.5	34.7	1.79	0.226
NX3 AIO 3MON(Nexus 3All In One 3-month group)		32	38	8.2	1.8	0.5	27.5	1.32	0.478
NX3 SOL IM (Nexus 3 Solo Plus immediate	16	32	0	30.5	2.3	7.3	52.4	3.46	0.171
NX3 SOL 3MON(Nexus 3 Solo Plus 3-month group)		32	3	13.4	2.0	0.5	41.6	2.53	0.308

TABLE III

Weibull parameters

Group	Characteristic strength	Modulus
NX2 AIO IM	16.5	3.7
NX2 AIO 3MON	3.5	3.5
NX2 SOL IM	2.5	1.2
NX2 SOL 3MON	1.4	1.9
NX3 AIO IM	6.1	4.4
NX3 AIO 3MON	3.8	2.1
NX3 SOL IM	31.9	5.9
NX3 SOL 3MON	12.6	3.3

Table IV

Significance level for groups[a]

NX3 SOL IM

NX2 AIO IM

NX3 SOL 3MON

NX3 AIO IM

NX3 AIO 3MON

NX2 AIO 3MON

NX2 SOL IM

NX2 SOL 3MON

[a]Groups connected by lines were not significantly different for microtensile bond strength at a 5% significance level (so groups not connected by any lines are considered to be significantly different).

TABLE V

Mode of failure results*

Group	Cohesive in Resin	Mixed	Adhesive
NX2 AIO IM	16 (100)	0 (0)	0(0)
NX2 SOL 3MON	0 (0)	9 (100)	0(0)
NX2 SOL IM	0 (0)	10 (100)	0(0)
NX2 AIO 3MON	11 (92)	1 (8)	0(0)
NX3 AIO 3MON	10 (91)	1 (9)	0(0)
NX3 AIO IM	12 (100)	0 (0)	0(0)
NX3 SOL 3MON	15 (94)	1 (6)	0(0)
NX3 SOL IM	13 (81)	3 (19)	0(0)

*Failure Mode, N (%)

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DISCUSSION

MICROTENSILE BOND STRENGTH TESTING

In this study, the NX3 Solo Plus immediate group had the highest bond strength values (30.5MPa), and the lowest bond strength was for the NX2 Solo Plus three months group (3.2MPa). The first null hypothesis could not be rejected. As NX3 is a relatively new material in the market there are no published results on microtensile bond strength for this material, so that these results could not be compared with any previous work. The better results obtained with NX3 and Solo Plus may be due to the fact that manufacturers made changes in the chemistry with the NX3 resin luting agent to prevent incompatibility issues with adhesive systems. Much work has been done showing chemical incompatibility when dual-cured systems are used along with simplified adhesive systems.[42, 47, 48] This forced manufacturers to make changes in the composition of NX3 resin luting agent by eliminating the tertiary amines and incorporating a new proprietary amine-free initiating system.

Considering the adhesive cement combinations, NX3 performed better with Solo Plus, but with the All in One, the NX2 got a higher value when compared to NX3 (TABLE II). Thus, the second null hypothesis that the type of adhesive used will influence the bond strength could not be rejected. This result is not in agreement with previous studies.[2] In a pilot work done for this study, the results obtained were in the range of 20 MPa for the NX2 Solo Plus group. In this experiment, 16 teeth were taken for each adhesive cement combination, and four beams were retrieved from each tooth, two to be tested immediately, and two beams to be stored for three months. But in the pilot study, the number of teeth used to obtain the beams was not taken into consideration since teeth were lost in the saw during the attempt to retrieve the beams. If the beams that were lost in the saw had been taken into consideration, perhaps a lower bond strength result would have been obtained.

Another possible explanation may be that NX2 used in this research came in two dispensing modes: the dual syringe, and the two paste system of base and catalyst. The latter one used for this study and this difference may have led to the incorporation of air bubbles in the cement, and also with the hand-mixing technique, there is a higher possibility of an unequal base-catalyst ratio leading to a chemical imbalance in the cement system. The above may have resulted in a compromise of the adhesive-cement interface and could explain why many teeth debonded and beams were lost while in the Isomet saw. Premature debonding of specimens can also be attributed to non-uniform stress placed on the bonded interface during sectioning using the slow-speed saw, which may have caused vibrations in the specimen.[27] Another possible reason may be that the Solo Plus was used without the activator. So it may also be assumed that if a higher number of beams had survived to be tested, higher bond-strength averages would have been obtained.

Although the results for NX3 Solo Plus Immediate and NX2 All In One immediate groups did not differ statistically, there was a difference of 11 MPa between the two groups. It suggests that the combination of NX3 Solo Plus Immediate had a much better bond strength than NX2 All In One Immediate, and this difference in bond strength could be clinically significant.

In relation to the storage time, the immediate group performed better than the three-month group. So, the null hypothesis that the storage time would influence the bond strength could not be rejected. This is in accordance with other studies that have shown that during storage hydrolytic degradation occurs resulting in lower bond strength value.[49-51] After aging specimens of simplified adhesive resins in different storage media for six months, Carrrilho et al. reported decreases in mechanical properties and bond strength values.[9, 51, 52] An explanation for this reduction in bond strength values may be related to a permeation of water that occurs after polymerization. There are possible water channels that originate from the surface of the hybrid layer and then extend through the adhesive layer to reach the adhesive composite interface. These channels have been given the name of water trees by Tay.[9] The reduction in bond strength values is seen with the three-month group of NX3 resin luting agent (Figures 17, 18). Even if the incompatibility issues with the tertiary amines have been resolved, there still may be the presence of certain other mechanisms, such as hydrophilicity of the adhesives and hydrolytic degradation, which may have an effect on the bond strengths and contribute to their early failures.

The hydrolytic degradation in the three-month groups with NX3 and Solo Plus was found also in the immediate groups for NX3 All In One and NX2 All In One. The hydrolytic degradation with the All In One systems is immediate and occurs as soon as 24 hours (Figures 19, 20). Results show high evidence of degradation for all the groups considered in this investigation. These types of cement adhesive combinations in clinical situations should be used with this understanding.

ANALYSIS OF MODE OF FAILURE

NX2 Solo Plus immediate and NX2 Solo Plus at three months had a significantly higher percentage of teeth that failed (Table V) in the mixed mode (Figures 15, 17). This can explain the low bond strength results obtained in these groups. We can see long resin tags (Figure 26); but, the dentinal tubules are unplugged, and the tags have been pulled out of these tubules. This demonstrates that the resin tags do not significantly influence the bond strength.[53] The weakest interface with this kind of failure may be between the adhesive and the dentin and very small stress was enough to create a rupture in the resin-dentin bonds. This also can be attributed to water getting trapped in this interface and weakening the bond.[19]

When we compare the failure modes for the other groups, the NX3 with Solo Plus and NX2 All In One, the failures were almost all cohesive failure in resin (Figure 20). The bond of the adhesive-dentin interface was stronger, and the failure may have occurred within the adhesive, in between the adhesive and the luting agent, or within the surface of the luting agent. A minimal number of teeth (one to three) failed in the mixed mode in these groups. Although higher MTS results were obtained with NX3 Solo Plus, the SEM analysis revealed fractured water blisters in the three-month group (Figure 18). This was a two-step, total-etch system and although the process of etching was done separately, these self-priming adhesives did not have a hydrophobic layer separating the adhesive and the luting agent. This may have resulted in the adhesive acting as a semipermeable membrane allowing water to get to the interface between the adhesive and resin luting agent resulting in blistering and degradation of the bond. This was evident with only three months of storage.[47, 54-56]

Not all the beams were examined under the SEM, and limitations in instrumentation with the stereomicroscope made it difficult to distinguish whether the resin belonged to the adhesive or the luting agent. Therefore, those failures were grouped together as adhesive failure in resin.

SURVIVAL ANALYSIS

The survival analysis helps us to predict the probability of beams surviving during various stress levels. Davidson[57] in 1984 reported that a minimum of 17MPa is required at the resin-dentin interface to compensate for the polymerization shrinkage stresses. Santos-Daroz et al.[58] have reported that resin luting agents also have high polymerization stresses and can disrupt the bond between the dentin and resin luting agent or the restorative material. As there are no ideal bond strength values reported with resin luting agents and dentin, using the above values as a guide, and assuming that 17 MPa is the minimum bond strength required to withstand contraction stresses during polymerization, it can be shown that most of the experimental groups of the present study have failed below 17 MPa. Better performance was seen in the NX3 Solo Plus immediate group, which had 90-percent survival of beams byond 17 MPa, and also in the NX2 All In One intermediate group, which had 50-percent survival beyond 17 MPa. For all the other groups, more than 50 percent of the beams failed below 17 MPa (Figure 14). The ability of these materials to withstand the oral stresses for a long duration may be questionable.

SUMMARY AND CONCLUSIONS

Sixty-four non-carious, non-restored human molar teeth were collected under an IUPUI/Clarion IRB approved protocol. The occlusal surfaces of the crowns were sectioned to expose dentin using a low-speed saw with a diamond blade. The dentin surfaces were ground with a 340-grit SiC paper under de-ionized water flow. The absence of enamel was verified using a stereomicroscope. The 64 teeth were then divided into four groups of 16 teeth each. Each group was subdivided into an immediate and a three-month group. Four beams were obtained from each tooth; two beams were used for the immediate and two beams for the three-month group. The dentin surfaces were then treated with one of the two adhesive systems, the two-step, total-etch Optibond Solo Plus and the one bottle self-etch Optibond All in One (SDS Kerr). Premade resin composite discs of 2-mm thickness were used to build a height of 4 mm, and two cements, NX2 and NX3 (SDS Kerr) were used to lute the composite discs to dentin. The teeth were then sectioned using a non-trimming technique to obtain the beams. The beams were stored in distilled water until testing. The beams were then subjected to microtensile bond strength testing using a universal testing machine at a crosshead speed of 1mm/min. All beams were analyzed using the stereomicroscope. Random samples from each group were then analyzed under the scanning electron microscope.

The present investigation sought to determine the microtensile bond strength of two different resin luting agent combinations at two different time periods (immediate and three months). This investigation determined that NX3 Solo Plus was the best adhesive cement combination because the highest bond strength result was obtained with this group; the degradation was not observed immediately according to SEM analysis. However, after three months of storage, osmotic blistering was evident. This suggests that even though the incompatibility issues with tertiary amines may have been resolved for NX3 resin luting agent, there may be other mechanisms, such as water movement across dentin and permeability of the adhesive systems, which can influence the bond strength values.

NX3 and NX2 combined with the All In One adhesive system showed signs of early degradation. This is in accordance with previous studies done with one-bottle adhesive systems. This suggests that the one-bottle, self-etch systems have a higher hydrophilicity and a larger amount of acidic resin monomers that are required to demineralize the dentin. This can lead to problems of accelerated hydrolytic degradation and water entrapment in the adhesive-resin interface resulting in low bond strengths.

When the failure modes were analyzed, most failures with NX3 Solo Plus and NX3 All In One were adhesive in resin, suggesting that the interface between the adhesive system and the dentin was strong. The NX2 resin luting agent with the Optibond Solo Plus adhesive system had the lowest bond strength values and all beams failed in the mixed mode, suggesting that the weakest link was between the adhesive and resin.

No ideal bond strength values were reported with resin luting agents and dentin. Assuming that 17 MPa is the minimum bond strength required to withstand contraction stresses during polymerization, we have shown that most of the experimental groups of the present study have failed below 17 MPa. As the results indicate, all the null hypotheses were accepted.

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ABSTRACT

BOND STRENGTH EVALUATION OF TWO RESIN

CEMENTS WITH TWO ADHESIVES AND

ANALYSIS OF MODE

OF FAILURE

by

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Cementing of indirect restorations with resin cements generally requires the pre-treatment of dentin with an adhesive. When dual-cured or chemical-cured resin cements are used with these single-step adhesives, incompatibility issues exist. This has resulted in manufacturers making chemical changes in their products. Kerr Dental markets a new resin cement, Nexus Third generation (NX3), which utilizes a proprietary redox system different from the second generation of composite luting agent (NX2). The aim of this study was to evaluate microtensile bond strength and mode of failure of NX3 and NX2 with two different adhesive systems (total-etch and self-etch) after 1 week and after 3 months of storage. Methods: Sixty-four non-carious teeth were sectioned to

expose the dentin using a low-speed saw. Dentin surfaces were ground with 320-grit SiC paper. The adhesives Optibond Solo Plus (SOL), and Optibond All In One (AIO) were applied, and resin cements (NX2, NX3) were used to lute 4-mm composite discs to the treated dentin surfaces. Microtensile bond strength was determined at 1 week (IM) and after 3 months (3MON) of storage using a universal testing machine (MTS). All specimens were examined under the stereomicroscope to determine the mode of failure. Random specimens from each failure group were examined using scanning electron microscopy. Statistical Analysis: Comparisons between the treatment combinations for differences in microtensile bond strength were performed using Weibull-distribution survival analysis. Comparisons between the treatment combinations for differences in the failure mode were performed using Fisher's Exact tests. The group NX3 SOL IM (30.5 MPa) had significantly higher bond strength than NX3 SOL 3MON (13.4 MPa); NX3 AIO IM (11.3MPa); NX3 AIO 3MON (8.2 MPa; NX2 AIO 3MON (5.8 MPa); NX2 SOL IM (6.3 MPa), and NX2 SOL 3MON (3.2 MPa). The group NX2 AIO IM (19.3 MPa) was not significantly different from NX3 SOL IM. The group NX2 SOL 3MON and group NX2 SOL IM had a significantly higher percentage of teeth with mixed failure than all of the other groups. None of the other groups had significantly different failure mode. The group NX3 SOL IM had 90-percent beam survival beyond 17 MPa, and NX2 AIO IM had 50 percent of beams surviving beyond 17 MPa, a better performance. For all the other groups, more than 50 percent of beams failed below 17 MPa. Results show high evidence of degradation for all groups considered in this investigation. The use of these types of cement adhesive combinations in clinical situations should be used with this understanding.

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