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Shear Bond Strength of Three Different Nano-Restorative Materials to Dentin

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Clinical Relevance

Nano glass ionomer exhibited significantly lower shear bond strength compared to nano-composites. The self-etch adhesive showed higher shear bond strength than etch&rinse adhesive for both nanofill and flowable nanofill composites.

SUMMARY

Objectives: To evaluate the shear bond strength (SBS) of a nano-composite, a flowable nano-composite and a nano glass ionomer to dentin *in vitro*.

Materials and Methods: Sixty human molars were ground flat, exposing the dentin surfaces, and they were randomly divided into five groups according to the restorative materials and adhesive systems used (n=12/group). The restoratives were applied to all dentin surfaces according to the manufacturer's instructions, using a special jig (Ultradent) in the following manner: Group 1: a nano-composite (NC) (Filtek Supreme XT-3M ESPE) was applied with a two-step self-etch adhesive (SE) (Adper SE Plus-3M ESPE); Group 2: NC was applied with an etch&rinse adhesive (SB) (Adper Single Bond 2-3M ESPE); Group 3: a flowable nano-composite (FNC, Filtek Supreme XT Flow-3M ESPE) was applied with SE; Group 4: FNC was applied with SB and Group 5: a nanofilled resin-modified glass ionomer (Ketac N100-3M ESPE) was applied with Ketac Nano Primer (3M ESPE). The bonded specimens were stored in distilled water (37°C, 24 hours) and tested for SBS in a universal testing machine (1 mm/minute). Two specimens from each group were subjected to SEM evaluations of the adhesive interfaces. Failure modes were determined using a stereomicroscope. The mean SBS values were calculated and the data were analyzed with the Kruskal Wallis and Mann-Whitney-U tests (p<0.05). Results: Mean SBS values (MPa) for the groups were 13.64; 7.83; 11.20; 4.12 and 0.64 for Groups 1, 2, 3, 4 and 5, respectively. Group 1 exhibited a significantly higher value than all the other groups; whereas, Group 5 had the lowest value (p<0.05). The SE adhesive yielded higher bond values than the SB adhesive with NC

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and FNC restorative materials. Failure modes in all the groups were primarily adhesive. Conclusion: The results demonstrate the capacity of the current two-step self-etch adhesive to outperform the etch&rinse adhesive in conjunction with the two nano-restoratives tested.

INTRODUCTION

Nanotechnology, also known as molecular nanotechnology or molecular engineering, is the production of functional materials and structures in the range of 0.1 to 100 nanometers—the nanoscale—by various physical or chemical methods. The intense interest in using nanomaterials stems from the idea that they may be used to manipulate the structure of the materials to provide dramatic improvements in electrical, chemical, mechanical and optical properties.[1] In recent years, nanotechnology has been used in the composition of different types of resin composites. Recently, a resin-modified photo-polymerizable glass ionomer based on nanotechnology was introduced to the market, providing the benefits of improved surface polish and esthetics.

Numerous commercial bonding systems are available under two simplified approaches: "etch&rinse" and "self-etch" adhesives.[2] In the etch&rinse technique, acid is applied to enamel and dentin simultaneously, followed by rinsing and the application of a combined primer and adhesive resin.[3] With self-etch adhesive systems, the etching and priming of the dentin and enamel occur simultaneously by subjecting the restorative surfaces to acidic resin adhesives. Thus, critical procedures, including rinsing of the etchant and priming of the hydrated collagen fibers, are eliminated with self-etch systems.[4] However, these self-etch systems still need to be investigated to determine whether these materials can produce strong, durable bonds to tooth structure. The results of the effectiveness of self-etch adhesive systems when compared with etch&rinse adhesives are contradictory.[5-6] Some perform similarly between systems,[6-7] while others suggest a superiority of the etch&rinse materials.[5,8]

Strong, durable bonds between restorative materials and tooth substrate are essential, not only from a mechanical standpoint, but also from the biologic and esthetic perspectives. Good marginal adaptation of dental materials reduces microleakage, staining, pulpal irritation and recurrent caries.[9] The bonding of resin-based restorative materials to dentin has always been more challenging compared to enamel bonding. Dentinal bonding is believed to be more difficult and less predictable over time, mainly because dentin is a "vital" tissue with high water and organic content (vs enamel) and its microstructure is dominated by tubules.[10] In selecting an adhesive system for clinical use, bond strength and sealing ability should play major roles.

The current *in vitro* study evaluated the shear bond strength (SBS) of a nano-composite, a flowable nano-composite with two different adhesive systems (etch&rinse adhesive, two-step self-etch adhesive) and a novel nanofilled resin-modified glass ionomer to dentin.

METHODS AND MATERIALS

Table 1 presents the materials used in the current study. Sixty freshly extracted, unerupted human third molars were collected and stored in distilled water for up to one month. The teeth were rinsed in running distilled water for 30 minutes, then embedded in auto-polymerizing acrylic resin (Simplex Rapid, Kemmdent, Associated Dental Products Ltd, Wiltshire, UK) with their buccal surfaces positioned for surface treatment and bonding. After polymerization of the embedding resin, the surfaces were abraded, then sequentially polished in a polishing machine (Mecapol P230, Presi Tavernoles 38, Brieet Angonnes, France) using 400 grit and 600 grit silicon carbide paper with water cooling until a uniform layer of dentin was evident. The exposed dentin surfaces were inspected with a stereomicroscope (Leica MS5, Leica Microsystems, Singapore, Singapore) to ensure that no enamel remained and no pulp exposure had occurred. All the specimens were then randomly divided into five groups according to the adhesives and restorative materials used. They are listed below:

Group 1: The two-step self-etch adhesive (Adper SE Plus, 3M ESPE, St Paul, MN, USA) was applied by first treating the dentin with Liquid A, creating a continuous red-colored layer on the surface. Liquid B was then applied and scrubbed into the surface of the bonding area for 20 seconds. The red color disappeared quickly, indicating that the etching components had been activated. After the treated dentin surface was air dried thoroughly for 10 seconds to evaporate the water, a second coat of Liquid B was applied and lightly air thinned, then it was light cured for 10 seconds (Elipar FreeLight 2 LED Curing Light, 3M ESPE) (light output:1000mw/cm^2). Filtek Supreme XT (3M ESPE) was applied to the adhesive treated surface according to the manufacturer's recommendations.

Group 2: The dentin surfaces were etched with 35% phosphoric acid gel (Scotchbond Etching Gel, 3M ESPE) for 15 seconds, rinsed for five seconds and gently air dried. Then, two consecutive coats of Adper Single Bond 2 (3M ESPE) were applied to the etched surfaces, gently agitated for 10 seconds, thoroughly dried with air and light cured for 20 seconds. Filtek Supreme XT was applied, similar to Group 1.

Group 3: Adper SE Plus adhesive was applied as in Group 1. Filtek Supreme XT Flow (3M ESPE) was then applied according to the manufacturer's recommendations.

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Table 1: *Products Used in This Study*

Brand Name	Product Type	Composition	Application Procedure
Filtek Supreme XT 3M ESPE, St Paul, MN, USA	nanofill composite	Bis-GMA, TEGDMA, UDMA, bisphenol Apolyethylene glycol diether dimethacrylate, silica nanofillers (5-75 nm), zirconia/silica nanoclusters (0.6-1.4 µm)	Light cure for 20 seconds
Filtek Supreme XT Flow 3M ESPE, St Paul, MN, USA	flowable nanofill composite	Bis-GMA, TEGDMA, Bis-EMA, Non-agglomerated/non-aggregated 75nm silica nanofiller; non-agglomerated/non-aggregated nanofiller; non-agglomerated/non-aggregated 15-20 nm zirconia nanofiller and loosely bound agglomerated zirconia/silica nanocluster, consisting of agglomerates of 5-20 nm primary zirconia/silica particles	Light cure for 20 seconds
Ketac N100 3M ESPE, St Paul, MN, USA	light-curing nano-ionomer restorative	De-ionized water, HEMA, Vitrebond copolymer (a methacrylate modified polyalkenoic acid), fluoroaluminosilicate glass, nanomers and nanoclusters	Apply Ketac N100 primer for 15 seconds, gently air dry and light cure for 10 seconds. Adequately air dry Place Ketac N100 light-curing nano-glass ionomer restorative material, light cure for 20 seconds
Adper SE Plus 3M ESPE, St Paul, MN, USA	two-step self-etch adhesive	**Liquid A** Water, HEMA, Surfactant, Pink colorant **Liquid B** UDMA, TEGDMA, TMPTMA (hydrophobic trimethacrylate), HEMA phosphates, MHP (methacrylated phosphates), Bonded zirconia nanofiller, Initiator system based on camphorquinone	Apply Liquid A creating a continuous red-color layer on the surface Apply Liquid B, scrub into the surface for 20 seconds, air dry for 10 seconds, apply second coat of Liquid B, air dry, light cure for 10 seconds
Adper Single Bond 2 3M ESPE, St Paul, MN, USA	etch&rinse adhesive	Bis-GMA, HEMA, dimethacrylates, ethanol, water, a novel photoinitiator system and a methacrylate functional copolymer of polyacrylic and polyitaconic acids, silica nanofiller	Apply Scotchbond Etchant (35% phosphoric acid-3M ESPE) to dentin for 15 seconds and rinse for 5 seconds, gently air dry Apply two coats of Adper Single Bond 2, gently aggitate for 10 seconds, thoroughly air dry, light cure for 20 seconds

HEMA: 2-hydroxyethyl methacrylate
UDMA: urethane dimethacrylate
TEGDMA: triethyleneglycol dimethacrylate
Bis-GMA: bis-phenol A diglycidylmethacrylate
Bis-EMA: bis-phenol A polyethoxylated dimethacrylate

Group 4: Adper Single Bond 2 adhesive was applied, as in Group 2. Filtek Supreme XT Flow was added, as in Group 3.

Group 5: A light-curing primer (Ketac N100 nano-ionomer primer) was applied to the dentin for 15 seconds, gently air dried and light cured for 10 seconds. Ketac N100 was then applied to the primed surface according to the manufacturer's recommendations.

In all specimen preparations, after the adhesive application, the specimens were clamped in the Ultradent Bonding Jig (Ultradent Products Inc, South Jordan, UT, USA), and the bonding template, with an inner diameter of 2.3 mm and a height of 3 mm, was attached to the dentin surfaces. All the restorative materials were applied in two increments, and each increment was light cured for 20 seconds for the light-cured nano-ionomer restorative with the LED light-curing unit.

The bonded specimens were stored in distilled water at 37°C for 24 hours. Shear bond testing was performed using a knife-edge blade in a universal testing machine (LR50K, Lloyd Instruments Ltd, Fareham, Hants, UK) at a crosshead speed of 1 mm/minute. The shear bond strength values were calculated as the ratio of fracture load and bonding area and were expressed in megapascals (MPa). After shear bond strength testing, the fractured test specimens were examined under a stereomicroscope (16x) (Leica MS5, Leica Microsystems) and the type of failure (adhesive, cohesive or mixed) was recorded.

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Bond failure was characterized according to the area of resin remaining on the dentin surface.[8] Adhesive failures were characterized as having less than 25% resin composite remaining at the interfacial bond area. Cohesive failures were greater than or equal to 75% resin remaining at the interfacial bond area, and mixed adhesive/cohesive failures had 25% to 75% resin composite remaining at the interfacial bond area.

Two specimens from each group were kept for Scanning Electron Microscope (SEM) examination. The exposed cross-sectional interfaces of the restorative material/adhesive/dentin were sequentially polished with a series of silicone carbide abrasive papers from 600 to 2000 grit using running tap water as a lubricant. The polished surfaces were then etched with 10% phosphoric acid for 10 seconds, followed by deproteinization in 5% sodium hypochlorite for five minutes. After rinsing with distilled water, each sample was mounted on stubs, sputter coated with gold and examined under SEM (JEOL 6400, JEOL, Tokyo, Japan).

The mean and standard deviation of the SBS values were calculated. Normal distribution of the data and homogeneity of the variances were tested by the Shapiro-Wilk test and Levene's test, respectively. Since parametric test assumptions were not satisfied, the data were evaluated by the Kruskal Wallis test and the Mann-Whitney U-test with Bonferroni correction ($p<0.05$).

RESULTS

Table 2 shows the mean shear bond strengths (SBS) and standard deviations for the nano-restorative groups tested. Kruskal Wallis analysis revealed significant differences between the groups ($p<0.05$). Filtek Supreme XT used in conjunction with Adper SE Plus exhibited a significantly higher mean SBS value (13.64 ± 2.88 MPa) than the other groups. The nano-ionomer Ketac N-100 had a mean SBS value significantly lower than that of the other groups ($p<0.05$). Adper SE Plus presented a significantly higher mean SBS value than Adper Single Bond 2 for both nano-composite restorative materials ($p<0.05$). Filtek Supreme XT showed significantly higher SBS values than Filtek Supreme XT Flow with both Adper SE Plus and Adper Single Bond 2 ($p<0.05$).

Fracture patterns for each group are presented in Table 3. The most prevalent mode of failure in all groups was the adhesive mode. One cohesive failure was observed when Filtek Supreme XT Flow was used with Adper SE Plus. The cohesive failure mode was not observed in any of the other groups.

SEM photographs of nano-restorative materials bonded to dentin are shown in Figures 1 through 3.

Table 2: *Mean Shear Bond Strengths (MPa) and Standard Deviations (SD) of the Nano-restorative Materials Tested*

Groups	N	Mean (±) SD
1 Filtek Supreme XT-Adper SE Plus	12	13.64 ± 2.88[a]
2 Filtek Supreme XT-Adper Single Bond 2	12	7.83 ± 5.63[b]
3 Filtek Supreme XT Flow-Adper SE Plus	12	11.20 ± 4.12[b]
4 Filtek Supreme XT Flow-Adper Single Bond 2	12	4.12 ± 3.54[c]
5 Ketac N100	12	0.64 ± 0.60[d]

Significant differences in groups with the different superscript letter ($p<0.05$*).*

Table 3: *Failure Modes Per Experimental Groups*

Groups	Failure Mode		
	Adhesive	Cohesive	Mix
1 Filtek Supreme XT-Adper SE Plus	9	-	3
2 Filtek Supreme XT-Adper Single Bond 2	10	-	2
3 Filtek Supreme XT Flow-Adper SE Plus	8	1	3
4 Filtek Supreme XT Flow-Adper Single Bond 2	11	-	1
5 Ketac N100	8	-	4

DISCUSSION

The adhesion of restorative materials to dental substrates is a desirable property, because it is associated with prevention of material dislodgment and decreased marginal leakage.[9,11] Different mechanical tests have been proposed to assess the bonding performance of restorative materials.[7,12] Testing in shear mode is a relatively simple, reproducible and widely accepted test.[13-15] Although this mode of testing has been met with some criticism, it is still being used to evaluate the bonding potential of adhesives to dental structure.[12,16] The test may be particularly appropriate for testing glass ionomer cements, which present relatively low bond strengths and may not be suitable for other testing methodologies.[17]

The nano-restorative materials tested in the current study exhibited significant differences in SBS ($p<0.05$), even when tested with the same adhesive, meaning that the composite formulation also had an impact on bond strength. Materials containing more resin components in their composition may exhibit improved bond strength performance with evidence of mechanical interlocking.[18-19]

Resin-modified glass ionomer cements (RMGIC) represent a category of glass ionomer cement modified by the addition of monomer components, resulting in a hybrid materi-

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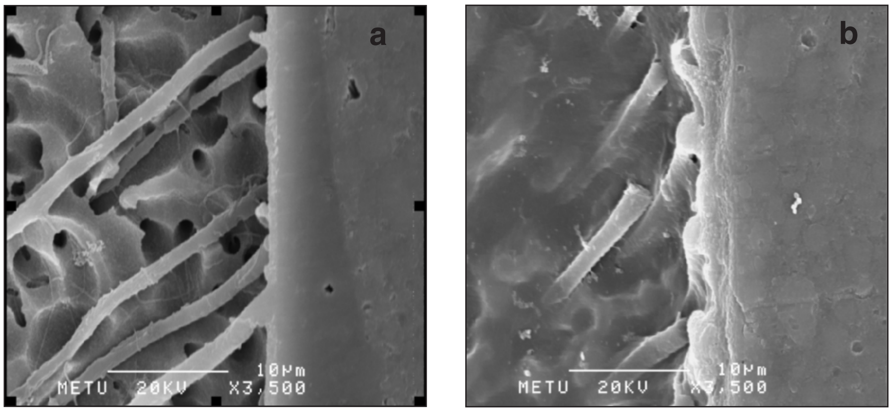

Figure 1: *SEM photomicrographs of Filtek Supreme XT/dentin interface: (a) used with Adper SE Plus, (b) used with Adper Single Bond 2, (original magnification 3500x). Note the resin tags formation with both adhesives. The tags seem longer for the self-etch adhesive (a); the etch&rinse adhesive shows more complete infiltration of the dentin (b).*

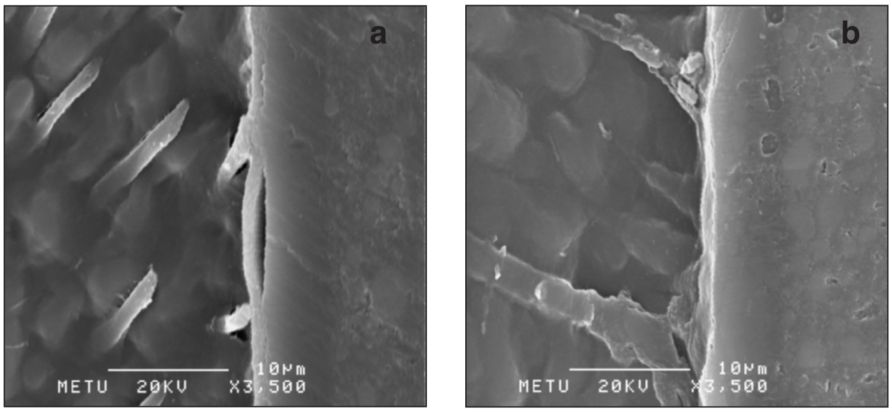

Figure 2: *SEM photomicrographs of Filtek Supreme XT Flow/dentin interface: (a) used with Adper SE Plus, (b) used with Adper Single Bond 2, (original magnification 3500x). Both photomicrographs show the formation of resin tags extending from the adhesive into the partially-dissolved dentin.*

al. RMGICs have been developed in an attempt to enhance the handling characteristics of conventional glass ionomer cements and increase the working time. These modifications also resulted in improved physical properties.[20] The new nano-ionomer material (Ketac N100) did not yield as high a mean SBS to dentin as other materials in the current study. As it was only applied following the primer application without any intermediary bonding material, this may have accounted for the relatively low bond strengths obtained.

Flowable composites were developed principally to provide their own unique brand of handling characteristics, rather than for their physical properties or bonding performance.[21-22]

Flowable composites have filler sizes similar to that of hybrid composites, but they have lower filler concentrations.[21] Some studies showed that traditional composites exhibited superior performance in all mechanical properties tested in terms of compressive strength, flexural strength, radiopacity and toughness compared to flowable composites.[22-23] As expected, in the current study, the nanofill composite Filtek Supreme XT exhibited higher SBS values than Filtek Supreme XT Flow with both adhesive systems.

Bond strength could also be affected by the mechanical properties of resin-based composites. Hasegawa and others[24] measured the mechanical properties of eight resin-based composites and correlated those properties with bond strength. In the current study, a nanofill composite, Filtek Supreme XT, was used. Thomsen and Peutzfeldt[25] compared the SBS of Filtek Supreme with the other four composites and revealed that a hybrid composite, Clearfil AP-X, results in the highest bond strength.

The bonding of resin composite to enamel and dentin is obtained through the use of an adhesive system. *In vitro* and *in vivo* studies have found the efficacy of adhesive systems differ greatly among the various types and brands of systems.[2,26] The efficacy has been shown to depend on numerous factors, such as infiltration of the adhesive into the demineralized tissue,[27-30] the degree of conversion and strength of the adhesive,[31-32] the mechanical properties of the resin composite[33] and compatibility between the resin composite

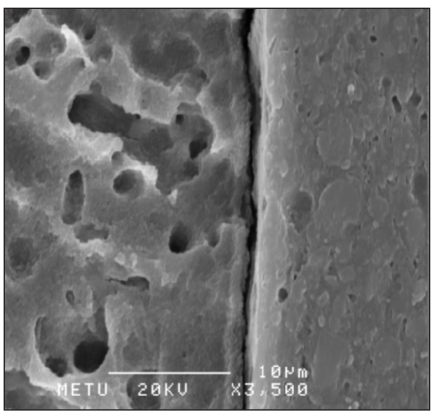

Figure 3: *SEM photomicrographs of Ketac N100/dentin interface (original magnification 3500x). Note the absence of the cement extension into the tubules.*

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and adhesive-treated dentin surface as regards sensitivity of the resin composite initiator system to an acidic environment[34] and surface energy parameters.[35] In the current study, the two-step self-etch adhesive system Adper SE Plus presented significantly higher SBS values than the etch&rinse adhesive system tested for both nano-composites. The superior bond mediating capacity of the two-step self-etch systems to dentin corroborates the findings of previous *in vitro* and *in vivo* studies[2,26,36-38] and is believed to result from a number of factors, one of the most important being the simultaneous demineralization and infiltration of dentin. These two simultaneous actions lead to a shallow but uniform resin-infiltrated dentin layer through which the residual hydroxyapatite remains available for chemical reaction.[2,39] In addition, the newer self-etch adhesive system tested has a color-change indicator, confirming the adhesive coverage and activation and making the procedure more precise.

Some studies have evaluated the importance of surface preparation method on bond strength[39-40] and have attempted to define the most clinically relevant smear layer preparation for use in *in-vitro* tests. The preparation of the specimen's surface with a bur *in vitro* is complex and time consuming and may be too difficult to standardize.[41-42] In most *in vitro* studies, such as the current study, dentin surfaces were prepared with 600-grit abrasive paper.[43-45]

An adhesive failure mode was predominantly observed in all the groups tested. Few specimens in each group exhibited a mixed failure pattern. The cohesive failures of adhesive materials are related to the high values of bond strength, predicting an effective bonding.[46] It is also suggested that the number of cohesive failures of adhesives indicate a normal distribution of stresses during the mechanical testing of bond strength.[47]

Resin-dentin interface analysis for the etch&rinse adhesive system revealed a thick, well-defined hybrid layer. On the other hand, the self-etch system presented a thin hybrid layer, as observed by Senawongse and others.[6] For both composites, Adper SE Plus showed longer resin tags and lateral tags compared to Adper Single Bond 2. Even though the dentin-resin interface analysis was performed on a small number of samples with an illustrative character, a thin hybrid layer was observed for Adper SE Plus, despite its high bond strength values.

Glass ionomer cements are sensitive to dehydration.[48] The true resin-modified glass ionomers are also prone to this phenomenon. In the current study, cracks seen in Ketac N 100 developed during the dehydration process for SEM evaluation may be the reason for separation of the cement layer from dentin.

Several studies reported that resin-modified glass ionomer-based materials exhibit superior retention to tooth structure compared to other adhesive strategies (for example, etch&rinse and self-etch adhesives) used with resin composites, although *in vitro* studies had shown that their bond strengths were lower than resin-based adhesives.[2,26,49]

Manufacturers recommend the use of dentin adhesives and composite of the same manufacturer to achieve the maximum effect of dentin bonding procedures, because differences in the chemical composition might lead to unexpected chemical reactions that are hazardous to bonding.[37] In the current study, all composites and adhesive systems were from the same manufacturer.

In recent years, the placement of resin-based direct composites has become a routine, well-established dental procedure. Despite the long-term results obtained with amalgam restorations, speculation about the possible health risks associated with mercury and the demand for esthetic restorative materials has contributed to the increased use of resin composites in posterior applications.[50] Nano composites are considered to be effective restorations in posterior teeth, with a number of long-term controlled clinical studies.[51-52]

The SBS values found in the current study were relatively lower than those reported in other bond strength tests. This discrepancy could be explained by differences in testing the conditions, the variable nature of dentin and operational factors.[53] Furthermore, regarding the effect of operator variability with different types of adhesive systems, it was found that technique sensitivity was one of the most important variables that affected optimal bonding.[54]

Another limitation of the current study was that no thermal cycling or artificial aging was performed to better simulate clinical conditions. Future studies are necessary to further evaluate the effectiveness and durability of nano-restorative materials.

CONCLUSIONS

Based on the findings of the current study and within the limitations of an *in vitro* investigation, it may be concluded that:

1) The highest bond strength was recorded with the nanocomposite when the self-etch adhesive was used.
2) For both composites, the self-etch adhesive exhibited statistically significantly higher SBS values than the etch&rinse adhesive.
3) Nano-ionomer cement presented the lowest bond strength compared to composites.
4) Failure mode analysis revealed a predominant adhesive mode for all the nano materials tested.

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