Comparative evaluation of bond strength of three contemporary self-etch adhesives: An *ex vivo* study

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Abstract

Aim: This study evaluated the effect of 2-hydroxymethyl methacrylate (HEMA) and the type of solvent on the tensile bond strength of the following three self-etch adhesives: Adper easy one (HEMA-rich adhesive) which contained ethanol, G-Bond (HEMA-free adhesive) which contained acetone, and Xeno V (HEMA-free adhesive) which contained butanol as a solvent. **Material and Methods:** Intact mandibular molars were mounted in self-cured resin and the occlusal surfaces were ground with # 600 SiC paper. Adhesives were applied on the prepared dentinal surfaces and the resin composite was condensed in the split brass mold (5 × 3 mm) placed over the adhesive surface. The specimens were stored in normal saline and placed in incubator at 37ºC. After 24 hours, the specimens were tested in tensile mode at a crosshead speed of 1 mm/min. Statistical analysis was done using One way ANOVA and Tukey's HSD test. **Results:** The mean bond strengths of Adper easy one, G-Bond, and Xeno V were 12.41 MPa, 10.09 MPa, and 8.67 MPa, respectively. **Conclusions:** Comparison of contemporary adhesives in this *ex vivo* study revealed that the ethanol-based HEMA-rich self-etch adhesive is better than HEMA-free self-etch adhesive that contained acetone and butanol as the solvents, when compared in terms of bond strength.

Keywords: Self-etch adhesive, solvent, tensile bond strength

Introduction

The demand of esthetics among the patients has led to the introduction of tooth-colored materials. Nowadays, composites are the most promising tooth-colored materials that have replaced silicates and acrylics. Composite resin bonds to enamel by means of acid-etching and to dentin with the help of dentin bonding agents.[1] Adhesion to dentin is not as reliable as adhesion to enamel, because of the morphologic, histologic, and compositional differences between them. To overcome the challenges in dentin bonding, various advancements have been made.[2]

The *ideal bonding system* should be biocompatible, should have sufficient bond strength to resist failure as a result of masticatory forces, bond indifferently to enamel and dentin, and be easy to use.[3] In the search of the ideal bonding agent, various dentin bonding systems were introduced in market but were more complicated and time consuming as they involved various clinical steps. During the past few years, the trend has been to move one step further by combining the etching, priming, and bonding with an attempt to develop fifth-, sixth-, and the seventh-generation adhesives.

The seventh-generation adhesives are the acidic primers and the rationale behind these systems is to superficially demineralize dentin and to simultaneously penetrate it to the depth of demineralization with monomers that can be polymerized in situ.[4,5]

The aim of this study was to compare the tensile bond strength of three seventh-generation self-etch adhesives, because higher the actual bonding capacity of an adhesive, the longer the restoration will survive *in vivo*.[6] Although clinical trials are the ultimate tests for the dental restorations, they cannot differentiate the true reason for failure due to the simultaneous impact of diverse stresses on the restoration, whereas laboratory testing can evaluate the effect of single variable while keeping the other variables constant. Thus, this study was planned to be conducted *ex vivo*.

Materials and Methods

The self-etch adhesives used in the study are listed in Table 1. Sixty recently extracted intact caries-free permanent mandibular molars were collected, cleaned of debris, blood, and calculus, and were then stored in the normal saline. The teeth were drilled in the deepest part of the central fossa

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Table 1: Materials used in the study

Self-etch adhesives	Manufacture	Code	Manufacturer
Adper easy one	3M ESPE, St Paul, MN,USA	EB	HEMA, Bis-GMA, Methacrylated phosphoric esters, 1,6 hexanediol methacrylate, Vitrebond copolymer, finely dispersed bonded silica with 7nm filler particles, ethanol, water, Initiators based on CQ, Stabilizers.
G bond	GC Corp, Tokyo, Japan	GB	4-META, UDMA, TEGDMA, acetone, distilled water.
XENO V	Dentsply, DeTrey, Germany	XV	Bifunctional acrylates, Acidic acrylate, Functionalized phosphoric acid ester, acrylamido 2-methyl propanol-2 sulfonic acid, Water, Tertiary butanol Initiator, Stabilizer

*Information provided by manufacturer, HEMA: 2-hydroxymethyl methacrylate, Bis-GMA: Bisphenol-A glycidyl dimethacrylate, 4-META: 4-Methacryloxyethyltrimellitate, UDMA: Urethane dimethacrylate, TEGDMA: Triethylene glycol dimethacrylate, CQ: Camphorquinone.

of the occlusal surface of each tooth with the help of round diamond bur (SF 21, Prime and Dental, Mumbai) up to the depth of 1.5 mm in order to standardize the depth of cavity. All the teeth were ground on the orthodontic trimmer until the drilled hole depth to expose the flat dentinal surface. This was followed by polishing of the flat dentinal surfaces with 600 grit silicon carbide paper (3M Products) to produce a uniform smear layer. After finishing, the teeth were then stored in the normal saline at room temperature.

The teeth were then placed on the glass slab with the flat dentinal surfaces facing the glass slab and stabilized at the periphery with the wax sheet. Rectangular aluminium molds of dimension 2.5 × 2 × 2.5 inches were placed in the position over the wax sheet bearing the teeth. A thin mix of self-cure acrylic resin (DPI Products) was placed in the molds to embed these teeth. A piece of adhesive tape was firmly attached on the occlusal surface to define the area of bonding. All the self-etch adhesives were applied according to manufacturer's instructions on the specified area of the tooth.

Group 1: Adper Easy Bond (3M ESPE, St Paul, MN, USA) was applied as one coat on the dentinal surface, left undisturbed for 20 seconds, dried with a strong blast of air for 5 seconds, and was further light cured for another 10 seconds.

Group 2: G Bond (GC Corp, Tokyo, Japan) was applied as one coat on the dentinal surface, left undisturbed for 5 seconds, dried with a strong blast of air for 5 seconds, and was further light cured for another 10 seconds.

Group 3: Xeno V (Dentsply, DeTrey, Germany) was applied as one coat on the dentinal surface, left undisturbed for 20 seconds, dried with a strong blast of air for 5 seconds, and was further light cured for another 20 seconds.

A split brass mold of diameter 3 mm and height 5 mm was used to form and hold the restorative resin onto the dentin surface. The resin composite was condensed into the mold in three equal increments. After the placement of first increment, a stainless steel wire loop was placed so that the composite stabilized it perpendicular to the surface and each increment was light cured for 20 seconds. The split brass mold and the adhesive tape were removed from the specimen 10 minutes after irradiation.

The bonded specimens were stored in normal saline and placed in the incubator at 37^{O}C and after 24 hours, the specimens were removed from the incubator and tested in tensile mode in Universal Testing Machine (Lloyd's Machine). The equipment was adjusted to operate at 1 mm/min, as different cross head speeds may influence the bond strength values.

For the tensile bond strength measurement, the wire protruding out of the composite cylinder was gripped into the superior cross head and the aluminium mold was held in the inferior cross head of the Universal testing machine. Tensile loading was done until the dislodgement of the composite cylinder from the dentinal surface occurred. The breaking load was measured and the results of the debonding force were tabulated in the values of force (Newton, N). The tensile bond strength was calculated by dividing the debonding force (N) by the debonding area (mm^2).

The mean values of bond strength were calculated for each experimental group and the data were analyzed by one way analysis of variance (ANOVA). Post hoc comparisons were carried using Tukey's test with the statistical significance set at $\alpha = 0.05$.

Results

The mean values of tensile bond strength and the standard deviations are shown in Table 2. For GROUP I (Adper Easy One), the bond strength measured was 12.41 MPa, whereas the mean tensile bond strength obtained for GROUP II (G-Bond) was 10.09 MPa and that obtained for GROUP III (Xeno V) was 8.67 MPa.

Means and standard deviation of the three groups were calculated as debonding force is a continuous dat. To compare all these three groups, one way ANOVA was applied. As *P* values came out to be statistically significant in all the three groups, Tukey's b test was applied to see multiple comparisons. All tests were 2 sided and alpha levels were taken at 0.05. Post hoc tests and Tukey's HSD test were applied to evaluate the difference in the mean bond strength of the experimental groups statistically.

Table 2: Mean debonding force and mean tensile bond strength of each experimental group

Group	Mean debonding force (N)	Mean bond strength (MPa)
Group I	87.8	12.41
Group II	71.4	10.09
Group III	61.34	8.67

The comparisons revealed differences in the mean bond strength of three self-etch adhesives containing different solvents and the difference between all the three groups was statistically significant ($P<0.05$).

Discussion

The concept of adhesive restoration has been essentially the most noteworthy development in ever progressing science. Thus, during the evolution of dentin bonding agents, attempts were made to achieve good bonding with enamel and dentin under difficult oral conditions and to ease out the technique. In this process, the recent development is combining etchant, primer, and bonding agent in one component to form seventh-generation dentin bonding agents which are commonly known as "*self-etch adhesives.*"[7] These self-etching adhesives can be used to etch both ground enamel and dentin simultaneously and they bond equally well to superficial and deep dentin.[8]

Ideally, the adhesives should be formulations based on hydrophobic monomers and high molecular weight, without additives such as solvents and water. However, due to the necessity for adhesive to penetrate into microporosities of the dentin, substrate inherently wet, hydrophilic resinous diluents, and solvents were incorporated into the adhesive. Thus, the liquid to be spread uniformly over a solid surface, the surface tensile of liquid must be less than the energy free surface of the substrate.[9] The low viscosity of primers and/or adhesive resins is partly due to the dissolution of the monomers in a solvent and will improve ability of diffusion into the microretentive tooth surface.[6] In fact, high values of bond strength to dentin, similar to those achieved in enamel, were obtained after the development of hydrophilic monomers and their association with organic solvents.[10] Also, during bonding, if the collagen network is allowed to collapse, the spaces between the collagen fibrils disappear and the adjacent fibrils come into intimate contact with each other. In the absence of water or other hydrogen forming substances, the collagen peptides may form intermolecular hydrogen bonds with the nearest neighboring collagen peptides which may contribute to the further collapse of the network by causing the shortening of the fibrils and leading to the increase in the stiffness. The addition of water and 2-hydroxymethyl methacrylate (HEMA) can break these intermolecular bonds with mass action, thereby softening the network and allowing it to re-expand.[11]

Thus, the present study was designed to *in vitro* compare the tensile bond strength of three recent seventh-generation bonding agents, HEMA-rich, HEMA-free, and containing three different solvents: Ethanol (Adper easy one, 3M ESPE), acetone (G-Bond, GC Asia, Corp), and butanol (Xeno V, Dentsply) which were bonded with the same microhybrid composite (Charisma, Heraeus Kulzer).

The study was planned to be conducted *in vitro* as it can help to evaluate the effect of single variable while keeping the other variables constant. The results of tensile bond strength measurement of three groups were tabulated and compared and the statistical analysis was done using one step ANOVA test and Post hoc Tukey's HSD test. The results showed that the GROUP I (Adper Easy one) with tensile bond strength of 12.41 MPa performed better than the other two groups; GROUP II (G-Bond) with bond strength of 10.09 MPa and GROUP III (Xeno V) with bond strength of 8.67 MPa. Adper Easy one showed higher tensile bond strength than G-Bond and Tetric N-bond self-etch, and the result was statistically significant ($P<0.05$). This result could be attributed to the composition of these dentin bonding adhesives. The inferior bond strength of G-Bond and Xeno V in comparison with Adper Easy one can also be contributed to absence and presence of HEMA. Adper Easy one contains HEMA (2-hydroxyl ethyl methacrylate) which is absent in case of G-Bond (HEMA-free) and also in Xeno V (HEMA-free). The hydrophilicity of HEMA makes it an excellent adhesion-promoting monomer and by enhancing wetting of dentin, HEMA significantly improves bond strength. In order to attain high bonding strengths to dentin, it is essential for the dentin substrates to have good penetrability and diffusibility.[12] In addition, the resin penetration into tubules can effectively seal the tubules and intertubular dentin and the resin infiltration can only occur if the mineral phase of dentin is removed by acidic conditioners. However, acidity of conditioner not only removes mineral from the dentinal matrix, but may also protonate collagen, changing the charges on the peptides and extracting noncollagenous proteins. However, once collapsed, it can be re-expanded with hydrophilic monomers such as HEMA. The higher bond strength following HEMA may be due to the fact that the demineralized collagen is kept wet and does not collapse as much as dentin that is dried with air blast. Also, the moist dentin may permit a more porous collagen network, which permits greater infiltration of adhesive monomers than do surfaces that are air dried and hence collapse.[13]

Furthermore, Adper Easy one contains ethanol as a cosolvent as compared with acetone present in case of G-Bond. Ethanol is a polar solvent that will form hydrogen bonds with its solutes. Ethanol removes water from these spaces causing the hydrogel to collapse, thus enlarging the interfibrillar spaces and allowing more resin infiltration.[14] It is speculated that the significantly higher bond strengths obtained when ethanol was used may be due to its Hansen's triple solubility parameters of the hydrogen bonds which is 19.4 $(J/cm^2)^{1/2}$ and is higher than that of dried collagen, 18.2 $(J/cm^2)^{1/2}$. The higher Hansen's triple solubility parameter of ethanol allows dehydration and stiffening of the matrix without allowing interpeptide H-bonding to collapse it.[15] Acetone has a high dipole moment and forms much lower hydrogen bonds due to which it is not able to expand the shrunken demineralized collagen. It has a high vapor pressure of 184 mm Hg at 20°C as compared with that of ethanol (43.9 mm Hg at 20°C). As the solvent evaporates, the viscosity of the bonding system increases, which decreases the ability of the bonding system to penetrate around the exposed collagen fibers and the opened dentinal tubules producing poor and incomplete hybrid layers.[16] Also, acetone has a boiling temperature of 56.5°C as compared with that of ethanol (78.3°C), which requires careful observation and handling of the product if the optimum ratio among the components is to be maintained.[17]

When G-Bond and Xeno V were compared with each other, it was observed that GROUP III (8.67 MPa) exhibited lower tensile bond strength as compared with GROUP II (10.09 MPa) and the result was statistically significant ($P<0.05$).

This result can be attributed to the fact that Xeno V has the lowest pH among the three self-etch adhesives. "Mild" self-etching appears most promising, especially with regard to bond stability. G-Bond and Adper easy Bond both have pH >2 which makes them "mild" self-etch adhesives, whereas Xeno V has pH <2 which makes it "intermediatory strong" self-etch adhesive.[6] In the light of bonding durability, mild self-etch adhesives have unique property that all hydroxyapatite are not removed from the interaction zone, and much calcium is available for additional chemical interaction with specific adhesive monomers. So, the bonds are stable, even in the aqueous environment and the mechanism is supposed to prolong the clinical lifetime of the restorations.[18] Also, the preservation of hydroxyapatite around the collagen in mild self-etch adhesives may protect the collagen against hydrolysis and thus prevents the early degradation of the bonds.

Conclusions

Under the circumstances of the present *ex vivo* study, Adper Easy one showed highest tensile bond strength when compared with the other two seventh-generation bonding agents: G-Bond and Xeno V. Thus, comparison of contemporary adhesives revealed that the HEMA-rich and ethanol-water-based self-etch adhesives are the "golden standard" in terms of adhesion durability. A further understanding of factors that contribute to the durability of adhesives and their bonding characteristics is needed.

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