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COMPARATIVE EVALUATION OF SHEAR BOND STRENGTH AND FAILURE PATTERN OF THREE DIFFERENT DENTIN ADHESIVE SYSTEMS - AN IN VITRO STUDY

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Abstract -Traditionally dentin bonding agents consist of separate components of etchant, primer and adhesive. Advancements in the field of adhesive dentistry have been aimed at reducing technique sensitivity by introducing single-bottle or all-in-one dentin bonding adhesive systems. The purpose of this study was to evaluate the shear bond strength of three adhesive systems [(G-Bond (GC), Xeno V+ (Dentsply) and Single bond universal (3M)] in regards to buccal surfaces and dentin depth.

Introduction - The success of a dental restoration depends on the high adhesive property of a material. Many restorative materials are available such as glass ionomer cement, composite and pit and fissure sealant which utilize the adhesive property of the material. Composite resins provide the best esthetics for anterior as well as posterior restorations.[1]

Modern adhesive dentistry allows the conservation of hard tissue to facilitate effective and efficient restoration. The goal of adhesive dentistry is to obtain an adequately strong bonding of restorative resin to the tooth for appropriate retention, reduced microleakage and thus providing superior color stability and clinical longevity of restoration.[2]

The adhesive bonding mechanism to dentin has been studied extensively. When the adhesive penetrates intratubular and intertubular dentin effective interlocking is established. Resin penetration into the conditioned dentin forms intratubular resin tags and a hybrid layer. The most important mechanism of resin adhesive to dentin is micromechanical attachment. Various factors such as acid-etching, moisture conditions of substrate and adhesive and dentin depth affect hybrid layer and resin tag formation.[3]

Self-etch primers were introduced to simplify the bonding procedures and prevent discrepancies between acid-demineralized dentin depth and penetration of primer to this demineralized layer. These primers etch through smear layers into the underlying dentin. They simultaneously condition, demineralize and infiltrate both enamel and dentin. Rinsing is not advised and the smear layer is changed but not eliminated. As etching and bonding are done by acidic monomers, the depth of demineralization equals the depth of monomer penetration which results in decreased chances of post-operative sensitivity.[4]

Self-etch adhesives are available as either 2-step or 1-step depending on whether self-etch primers and adhesives are provided separately or combined in a single solution. Self-etch adhesives have become popular, especially for their ease of use and faster application.[5]

Adhesive infiltration into the dentin and the thickness of the adhesive layer are correlated to rheological and chemical characteristics, but they can also be influenced by the mode of application. Various clinical approaches have been proposed to improve monomer infiltration. Improved bonding has been achieved by modifications like the use of an additional layer of hydrophobic resin agent, multiple-layer application, enhanced solvent evaporation and prolonged curing time intervals.[6]

An aqueous solution of acidic functional monomer is found in self-etch primers and adhesive systems whose pH remains comparatively higher than phosphoric acid etchants. It also contains Hema monomer which increases the wettability of dentin. To provide strength to the cross-linking formed from monomeric matrix bi- or multi-functional monomers are added.

The etching aggressiveness of self-etch adhesive systems depending upon acid dissociation constants can be classified into- “strong” (ph<1), “intermediately strong” (ph≈1.5), “mild” (ph≈2) and “ultra-mild” (ph≥2.5). Strong self-etching dissolves nearly all smear layers at dentin but does not remove the dissolved calcium phosphates. Partial removal of the smear layer occurs in case of mild self-etching which forms a thin hybrid layer. A nanometer interaction zone is formed by ultra-mild self-etching by exposing the superficial dentin
collagen.[7]

In current times, the development of new products is occurring at an unparalleled rate. Various all-in-one adhesives are marketed nowadays but there is much to be added about their capacity to adhere to dental hard tissues. Bond strength testing is useful for understanding and predicting the clinical behavior of adhesives.

Aim - The purpose of this study was to evaluate and compare the shear bond strength and failure pattern of three currently available all-in-one adhesive systems.

Materials and method - 90 freshly extracted human permanent maxillary/mandibular molars were selected.

Inclusion criteria -
- Sound human permanent maxillary and mandibular molars
- Noncarious teeth

Exclusion criteria -
- Carious teeth
- Teeth with cracks, fractures or craze lines

Specimen preparation-
- The teeth were cleaned and stored in saline solution at room temperature until use. The roots of the teeth were removed using a diamond disc at the cementoenamel junction under sufficient water cooling to obtain the crown portion. The teeth were cut in a mesiodistal direction to obtain the two halves. The teeth were embedded in auto-polymerizing resin in a rubber mold. The buccal surface

of the teeth was trimmed to expose dentin. The samples were then stored in water for 24 hours at room temperature to ensure full hydration of the teeth.

- **Formation of groups –**

Now the teeth were divided into three groups according to the different adhesives applied: -

- **Group 1-** G-Bond (GC) was applied on the dried surface and left undisturbed for 5-10 seconds. Now it was dried thoroughly under maximum air pressure for 5 seconds. In the presence of vacuum suction. Visible light curing unit was used to cure the adhesive.
- **Group 2-** Xeno V+ (Dentsply) was applied to wet the surface uniformly. Then the adhesive was gently agitated for 20 seconds. Forceful air was continued for 5 seconds or no more movement of the adhesive. Curing was done for 10 seconds.
- **Group 3 -** Single bond universal (3M) was applied and rubbed for 20 seconds. A gentle stream of air was directed for 5 seconds until the adhesive no longer moved and the solvent had evaporated completely. Now cured using curing light for 10 seconds.
- **Composite resin build-up -** A composite resin cylinder (3 mm diameter×2mm height) was built upon the adhesive layer using a plastic matrix and cured according to the manufacturer's instructions. A light-cured unit was used for 40 seconds at a light intensity of 1000 MW/cm2. The plastic matrix was removed by slitting it with a Bard Parker blade along its length after the curing of the composite. The specimens were then stored in saline at room temperature for 24 hours.
- **Thermocycling -** The specimens were then thermocycled for 500 cycles between 5˚c to 55˚c water bath. A dwell time of 30 sec was used for each bath.
- **Shear bond strength testing -** The specimens were then subjected to shear loading using the universal testing machine. The shearing load was applied at a cross-head speed of 1 mm/ min until bonding failure occurred. The shearing force was noted and shear bond strength was calculated as the ratio of fracture load and bonding area and expressed in megapascal units and the data was subjected to statistical analysis. After load testing, the type of failure was detected at ×10 magnification with a stereomicroscope.
- **Statistical analysis -** Data was entered in Microsoft Excel 2016 for Windows. Percentages, mean, standard deviation (SD), and minimum and maximum values of variables in different dentin adhesive systems were calculated.
- The Shapiro-Wilk test showed that shear bond strength values in different groups followed a normal distribution. Hence parametric test, one-way ANOVA was applied for comparison of shear bond strength between different groups.
- For comparison of failure mode between different dentin adhesive systems (nominal data), Pearson's chi-square test was applied. A P-value of less than 0.05 was deemed statistically significant. The statistical program for social sciences, version 21.0 (IBM corporation, Armonk, New York, USA), was used to analyze the data.

Observation and results-

Table 1: Comparison of shear bond strength between different dentin adhesive systems.

Dentin adhesive systems	Shear bond strength (mpa)	
	Mean ± sd	Min-max
G-bond	15.23 ± 3.90	12.03-30.89
Xeno v^+	16.36 ± 3.19	11.39-25.16
Single bond universal	17.60 ± 4.67	11.51-28.09
One-way ANOVA	F = 2.679, p = 0.074 (>0.05), not significant	

Figure 1: Comparison of shear bond strength between different dentin adhesive systems

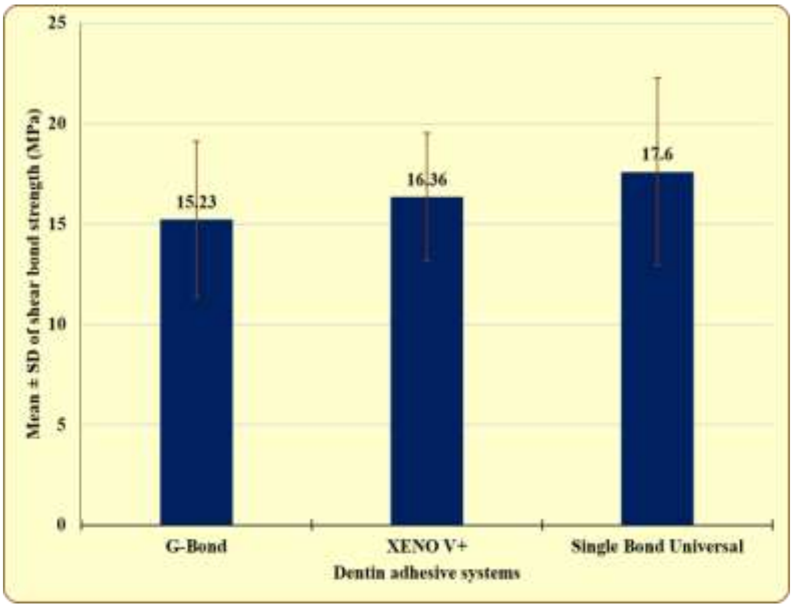

Table 1 and Figure 1 show a comparison of shear bond strength between different dentin adhesive systems.

1. Mean ± SD of shear bond strength in G-bond, Xeno v+, Single bond universal was 15.23 ± 3.90 Mpa, 16.36 ± 3.19 Mpa and 17.60 ± 4.67 Mpa, respectively.
2. Minimum and maximum values of shear bond strength in G-bond were 12.03 Mpa and 30.89 Mpa, in Xeno V+ were 11.39 mpa and 25.16mpa and in Single bond universal were 11.51 Mpa and 28.09 Mpa.
3. One-way ANOVA showed no significant difference in shear bond strength between different dentin adhesive systems (f = 2.679, p >0.05).

Table 2: Comparison of failure mode between different dentin adhesive systems.

Dentin adhesive systems	Failure mode			Total N (%)
	Adhesive N (%)	Cohesive N (%)	Mixed N (%)	
G-bond	22 (73.33)	00 (0.00)	08 (26.67)	30 (100.00)
Xeno v^+	20 (66.67)	00 (0.00)	10 (33.33)	30 (100.00)
Single bond universal	25 (83.33)	00 (0.00)	05 (16.67)	30 (100.00)
Total	67 (74.44)	00 (0.00)	23 (25.56)	90 (100.00)
Chi-square test	X^2 = 2.219, df = 2, p = 0.330 (>0.05), not significant			

Figure 2: Comparison of failure mode between different dentin adhesive systems

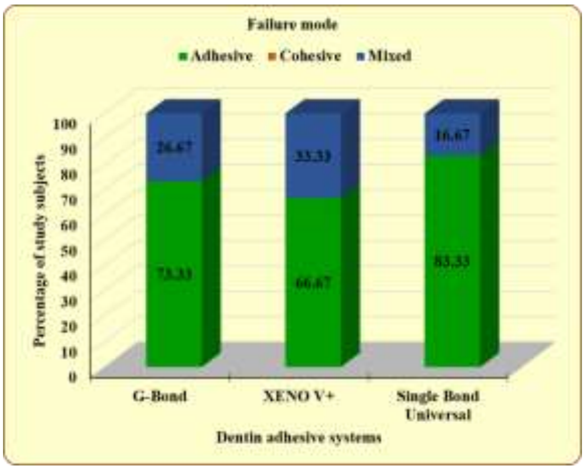

Table 2 and Figure 2 show a comparison of failure modes between different dentin adhesive systems.

1. In G-bond, 22 (73.33%) samples showed adhesive failure and 08 (26.67%) showed mixed failure.
2. In Xeno V+, 20 (66.67%) samples showed adhesive failure and 10 (33.33%) showed mixed failure.
3. In Single bond universal, 25 (83.33%) samples showed adhesive failure and 05 (16.67%) showed mixed failure.
4. None of the samples in any group showed cohesive failure. The chi-square test showed no significant difference between the groups for type of failure ($\chi2 = 2.219$, df = 2, p >0.05).

Discussion –

Adhesion in dentistry can be stated as the relation between bonding and stress. For a successful restoration, bonding should withstand the stress. The bonding to dentin has always been more challenging due to its heterogeneous nature. Therefore, in the present study, we aimed to determine the shear bond strength of three different adhesive systems to dentin. Strong and durable bonding between restorative material and tooth substrate is important for mechanical, biological and esthetic aspects.[8]

Dentin is a dynamic tissue. It is a biological compound of apatite crystals filled with collagen matrix. These crystals are dispersed between parallel micrometer-sized hypermineralized collagen-poor dentinal tubules containing peritubular dentin. It is composed of 50% minerals, 20% water and 30% organic matrix. As the dentin deepens the composition changes because superficial dentin has few tubules predominantly composed of intertubular dentin. This intertubular layer plays an important role during hybrid layer formation in superficial dentin.[1]

The exchange process by which inorganic tooth material is exchanged for synthetic resin is the principle of adhesion to tooth substrate. There are two phases: - The first is the removal of calcium phosphates leading to exposure of microporosities at the dentin surface. The second phase is hybridization which is the infiltration and polymerization of resin within the microporosities. This results in micromechanical interlocking due to the diffusion mechanism.[9] Proper demineralization of the dentin substrate, uniform resin impregnation and sufficient mechanical strength of the adhesive resin are important factors for creating a highquality resin/dentin interface for good dentin bonding. For a stable bonding self-etch adhesive should penetrate beyond the smear layer into the underlying dentin.[10]

The type of dentin, the amount of remaining humidity in the substrate, the application technique inherent to the adhesive system itself, chemical composition, type of diluents and dentin treatment influence the dentin bonding.[11] Some other variables are also there like intimate contact with dentinal tubules and lateral branches, thickness and mechanical properties of the bonding agents.[12]

Adhesive systems can be divided into two categories: total-etching and self-etching. The total-etch technique is based on the removal of the smear layer and demineralizing the dentin by acid etching and the self-etch system contains an acidic primer to demineralize the smear layer and subsurface dentin. There is a difference in bond strength of these two adhesive systems to tooth substrate.[13]

Van Meerbeek et al. classified contemporary adhesives based on the adhesion strategy and application procedure as 3-step etch&rinse adhesives, 2-step etch&rinse adhesives, 2-step self-etch adhesives, and 1-step self-etch adhesives.[14]

All the adhesive systems used in this study were self-etch bonding agents. The chemistry of self-etch systems is very challenging. Hydrophilic and hydrophobic monomers along with solvents and water are incorporated into a single bottle which makes these systems highly hydrophilic. The self-etch approach can be classified as a two-step or one-step application procedure.[15]

The drawbacks of the total-etch technique in etch–rinse adhesive systems are the risk of overetching, over-drying and over-wetting of dentin after the rinsing procedure, and also the use of multiple steps in the technique. This led to the development of two-step and then one-step self-etch adhesive systems.[16]

Incomplete polymerization and continued demineralization of the adjacent dentin structure in the tubules are some of the limitations of self-etch adhesives. The acidic formulation of allin-one adhesives has become more hydrophilic allowing deeper penetration. These adhesives penetrate the wet dentinal tubules deeply due to which the water content increases. Wang Y et al referred that this water acts as a major interfering factor in polymerization which leads to unpolymerized acidic and aggressive monomers to continue etching the dentin and leading to a detrimental impact on the bond.[17]

Self-etch systems are also classified according to acidity into – mild, moderate and strong. Strong self-etch adhesives can dissolve nearly all hydroxyapatite crystals. Mild self-etch adhesives have a pH of around 2 which can demineralize dentin only to a depth of 1 micrometer. Due to superficial demineralization residual hydroxyapatite remains partially attached to collagen.[7]

These bonding systems depend on partial demineralization of the dentin surface by acidic monomers which remove the smear layer and lead to exposure of collagen fibrils to penetration by resin monomers. The smear layer can affect the penetration of self-etch adhesives due to the neutralization of the acid monomers by buffering components.[18] Tay F R et al suggested that the bond strength of self-etch systems was not affected by smear layer thickness.[19]

Self-etch adhesives have been associated with less nano leakage. This has been attributed to resin impregnation occurring simultaneous to dentin etching. There is limited risk of discrepancy between the depth of dentin demineralization and hybridization which can be advantageous in the long term.[20]

The etch approach appears most promising regarding user friendliness and technique sensitivity.

G-Bond (GC) is a 7th-generation bonding agent composed of phosphoric acid ester monomer, 4 MET monomer, nano-filled particles, acetone and water solvents. It forms a nonconventional interface which is an advanced formulation. An insoluble compound is produced by this nano-level reaction for a better bond that is less likely to deteriorate from oral enzymes. Little or no exposure of collagen fibers is seen at the "nano interaction zone" which is extremely thin (only 300 nanometers). 5% nanofiller seals tubules which minimizes pulpal sensitivity, microleakage and microbial invasion.[21]

Xeno V+ (Dentsply) is a one-component self-etch adhesive. It has a high tolerance towards storage conditions and comprises bifunctional acrylate, acidic acrylate, functionalized phosphoric acid ester, water, tertiary butanol, initiator and stabilizer. The acidic monomer is added to increase the acidity. Acrylic acid acts as a wetting agent which promotes the penetration of bigger cross-linking monomers into tooth substrate.[22]

Single bond universal (3m espe) has a pH of around 2.7. The chemical bond formed between 10-MDP and dentin provides acidity for its self-etch property along with stable and durable interfaces. Excellent mechanical properties and a high rate of conversion of its filled hydrophobic resin have been shown by 10-mdp molecule. Thus the presence of mdp molecule in Single Bond Universal may explain higher shear bond strength. It also contains polyalkenoic acid copolymer (vitrebond copolymer) which may compete with mdp monomer for Ca-binding sites in hydroxyapatite. It can prevent monomer approximation during polymerization due to its high molecular weight.[23]

Different mechanical tests assess the bonding performance of restorative materials. Shear bond strength has been widely used to determine the bonding efficacy of adhesive systems to dental structures. The shear bond strength test is a simple procedure to evaluate the bond strength of dental adhesives. Thus in this study, shear bond strength testing was done with a universal testing machine which is convenient and popular to evaluate the binding ability of adhesive systems.[24]

According to Nair et al shear stress is considered to be more representative of the clinical situation for dentin bonding. The effectiveness of dentin and resin adhesive systems in clinical dentistry is largely dependent on their ability to maintain an intact bond. This is because strong marginal adaption helps to minimize microleakage, recurrent caries, and
pulpal irritation.[25]
The main objective of the bond strength test is to determine the bonding of an adhesive system to dental hard tissues when bonded to a composite. It has been stated that 17-20 mpa bond strength value is required to resist the contraction forces of resin composite materials. Self-etch adhesive systems depend on acidic monomers for simultaneous demineralization and infiltration of dentin. This acidity must be neutralized by the mineral content of the tooth structure for complete polymerization of the adhesive film.[26]
In the present study, the highest shear bond strength was observed for Single bond universal (3M) compared to Xeno V+ (Dentsply) and G-Bond (GC). The shear bond strength value obtained for Single bond universal in this study i.e. 17.60 Mpa is similar to the value obtained by Kumari R V et al and Ayar M K et al i.e. 17.31 Mpa and 17.90 Mpa respectively due to
the presence of 10-MDP molecule which has excellent mechanical properties.[1,27]
In the present study, Single bond universal (3M) had higher shear bond strength than Xeno V+ (Dentsply) but there was no significant difference between these two groups which is
similar to the study done by Cheema R et al.[28]
The lowest bond strength value in this study was obtained by the self-etching HEMA-free adhesive, G-bond. In a study by Sindhu S K et al, phase separation among adhesive compositions was confirmed, as droplets entrapped during solvent evaporation from HEMAfree adhesives. This phenomenon could be explained by solvent evaporation such as ethanol and acetone, which affected the balance of solvents and resin monomers and caused water to
separate from other compositions of the adhesive.[29]
Uppin V M et al also evaluated and compared the shear bond strength of different self-etch primers to dentin. The finding of G-Bond in their study i. e. 16.72 Mpa is similar to our study i.e. 15.23 Mpa.[4]
On the negative, Jaysheel A et al reported a shear bond strength value as low as 2.46 Mpa for Single bond universal which could be due to the presence of polyalkenoic acid copolymer which in combination with mdp has shown antithetical results. The polyalkenoic acid copolymer can compete with the mdp monomer in hydroxyapatite for Ca-bonding sites which could prevent monomer approximation during polymerization due to its high molecular
weight.[23]
Self-etching adhesive systems rely on simultaneous demineralization and infiltration of enamel and dentin by acidic monomers. The mineral content of the tooth structure neutralizes this acidity to allow complete polymerization of the adhesive film. The smear layer and dissolved minerals are removed during the rinsing step in total-etch adhesives. The hydrolytic stability of the self-etching adhesive systems remains unresolved because of some residual
acidity and the fact that the smear layer is not removed.[10]
The result of this present study showed that there was no significant difference in the mean shear bond strength among the self-etch adhesive systems tested i.e. G-Bond (GC), Xeno V+ (Dentsply) & Single bond universal (3M). Mean ± SD values of shear bond strength of GBond, Xeno V+ and Single bond universal were 15.23 ± 3.90 Mpa, 16.36 ± 3.19 Mpa and
17.60 ± 4.67 Mpa, respectively.
Failure patterns for each group are presented in Table 2. An adhesive failure mode was predominantly observed in all groups tested. The cohesive mode of failure was not observed in any of the groups. Few specimens in each group exhibited mixed failure patterns.
Thermocycling is a common method to simulate intraoral aging and stresses applied to the bonding interface and bond strength tests. In this study thermocycling for 500 cycles between 5°c-55°c was done with a dwell time of 30 seconds which is similar to the study done by
Kumari R V et al.[1]

Various factors such as type and age of teeth, degree of dentin mineralization, the dentin surface being bonded, type of bond strength test, storage medium, relative humidity in substrates, complex nature of testing procedures, sensitivity of manipulation of these systems and restorative materials influence the in-vitro bond strength of adhesive systems. These
variations could be responsible for the different values obtained in the present study.[30]

Conclusion -
Within the parameters of this study, the following conclusions were drawn: - 1)
Mean ± SD of shear bond strength of G-Bond was 15.23 ± 3.90 Mpa.
2) The mean ± SD of the shear bond strength of Xeno V^+ was 16.36 ± 3.19 Mpa.
3) Mean ± SD of shear bond strength of Single bond universal was 17.60 ± 4.67 Mpa.
4) The highest shear bond strength value was obtained by Single bond universal followed by Xeno V^+ and G-Bond. The shear bond strength of all three adhesive systems was comparable.
5) In G-Bond, 22 (73.33%) samples showed adhesive failure and 08 (26.67%) showed mixed failure, in Xeno V+, 20 (66.67%) samples showed adhesive failure and 10 (33.33%) showed mixed failure and in Single bond universal, 25 (83.33%) samples showed adhesive failure and 05 (16.67%) showed mixed failure. Adhesive failure was predominantly observed while some of the samples showed mixed failure. None of the samples in any group showed cohesive failure.

However, further investigations with additional in vivo and in vitro tests are desirable to evaluate the shear bond strength of different dentin adhesive systems to dentin.

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