Comparative evaluation of shear bond strength of sixth- and seventh-generation bonding agents with varying pH – An *in vitro* study

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**Abstract**

**Introduction:** To compare and evaluate the shear bond strength of sixth- and seventh-generation bonding agents with varying pH – an *in vitro* study.

**Materials and Methods:** Eighty extracted human premolar teeth were collected and cleaned and polished with pumice and water. The root portion of teeth was resected, and only the coronal portion was embedded in the cold-cure acrylic resin. The labial surface of mounted teeth was prepared with a high-speed handpiece using #245 carbide bur. The samples prepared were divided into four groups, with 20 specimens in each group:
- Group A: Sixth-generation bonding agent, Adper Prompt L-Pop (APLP) (3M ESPE)
- Group B: Sixth-generation bonding agent, Xeno III (X III) (Dentsply)
- Group C: Seventh-generation bonding agent, Adper Easy One (AEO) (3M ESPE)
- Group D: Seventh-generation bonding agent, Xeno IV (X IV) (Dentsply).

Tooth surface were rinsed and dried, and bonding agents were applied on tooth surface. Composite resin (Z-350 XT, 3M ESPE) was placed in a two-layer increment on tooth and was light cured. Specimens were subjected to the universal testing machine in a compression mode force at a crosshead speed of 1 mm/min keeping blade parallel to the adhesive–dentin interface. Shear force required to debond the specimen was recorded in megapascal. The data obtained were analyzed statistically using ANOVA and post hoc test.

**Results:** AEO (pH = 2.3, Group C seventh generation) showed higher bond strength, and pH values did not influence the shear bond strength significantly in the tested adhesive systems.

**Conclusion:** The pH values did not influence the shear bond strength significantly in the tested adhesive systems. ADPER EASY ONE (pH = 2.3, GROUP C Seventh Generation) showed higher bond strength followed by XENO IV (pH = 2.1, GROUP D) and XENO III (pH = 1.5, GROUP B) on dentinal surface, whereas ADPER PROMPT L POP (pH = 0.7 to 1 Sixth Generation, GROUP A) showed lower bond strength.

**Keywords:** Newer bonding agent; pH; shear bond strength

**INTRODUCTION**

Adhesive techniques for restoration of teeth have broadened the horizons of esthetic dentistry. In the initial era of restorative dentistry, retention of restoration often needed the removal of sound tooth structure to provide large undercutts and to gain auxiliary retentive aids.[1] The...
introduction of newer bonding systems in adhesive dentistry resolves this problem. Adhesive restoration reinforces the weakened tooth structure by effectively conducting the functional stresses across the bonding interface.[2,3]

Self-etch adhesives simplified the bonding procedures, were developed and to prevent discrepancies between the depth of dentin demineralize by the acid and the ability of primer to penetrate this demineralize layer,[3,4]

The self-etch adhesives use weaker acids that remove the smear layer partially and maintain the smear plugs and create thin hybrid layers.

These simplified systems are suggested to reduce technique sensitivity and shorten clinical procedures.[3,4]

The self-etch adhesive pH is higher than phosphoric acid. Self-etch adhesives are classified into three categories based on pH value: mild (pH of 2.5 or more) moderate (pH of approximately 1), and strong (pH <1) dissolve the smear layer completely and form a relatively thick transitional layer. Currently, several self-etching systems are available, but insufficient information is known about their capacity to adhere to dental hard tissues.

Since testing bond strength is used as a screening tool to help understand and predict the clinical behavior of adhesives, this in vitro study was designed to investigate and compare the shear bond strength to dentin using several self-etch adhesive systems with varying pH.

**MATERIALS AND METHODS**

Eighty extracted intact human premolar teeth were collected and cleaned, and then, the teeth were polished with slurry of pumice and water. The root portion of teeth was resected, and only the coronal portion was embedded in the cold-cure acrylic resin with the help of custom-made silicone rubber mold of dimensions 2 cm × 2 cm.

Teeth were then mounted horizontally, and labial surface was prepared with high-speed handpiece using #245 carbide bur.

The samples prepared were then divided into four groups with 20 specimens in each group, and for ease of identification purpose, each acrylic block was painted with different colors.

- Group A: Sixth-generation bonding agent, Adper Prompt L-Pop (APLP) (3M ESPE) Red
- Group B: Sixth-generation bonding agent, X III (Dentsply) Green
- Group C: Seventh-generation bonding agent, Adper Easy One (AEO) (3M ESPE) Yellow
- Group D: Seventh-generation bonding agent, X IV (Dentsply) Pink.

Tooth surface was then rinsed and dried, and bonding agents were applied on tooth surface. As shown in Figures 1-4.

Then, the composite resin (Z-350 XT, 3M ESPE) was placed in a two-layer increment on tooth in silicone rubber mold (2 mm × 2.5 mm) and was light cured (Woodpecker Led) for 40 s. The samples were thermocycled at a temperature 5°C–55°C at a dwell time of 30 s. In distilled water, all the specimens were stored for 24 h before shear bond testing. Specimens were subjected to the universal testing machine in a compression mode force at a crosshead speed of 1 mm/min keeping blade parallel to the adhesive–dentin interface. Shear force required to debond the specimen was then recorded. Debonding stress was calculated in megapascal (MPa) by the ratio of maximum load in newton to the surface area of prepared resin cylinder (Mpa + N/mm²). The data so obtained were then tabulated and analyzed statistically using ANOVA and post hoc test.

**RESULTS**

A significant difference was noted in the shear bond strength to dentin of four self-etch adhesives tested. The pH value did not influence the shear bond strength significantly in the tested adhesive systems. ADPER EASY ONE (pH= 2.3, GROUP C Seventh Generation) showed higher bond strength followed by XENO IV(pH = 2.1, GROUP D), XENO III (pH = 1.5, GROUP B) on dentinal surface, where as ADPER PROMPT L POP (pH =0.7 to 1 Sixth Generation, GROUP A) showed lower bond strength. Based on this result, it appears that seventh generation is beneficial than sixth generation in dentin bonding as it has fewer steps and higher shear bond strength, as shown in Tables 1 and 2. As shown in Graphs 1 and 2.

**DISCUSSION**

Dentin is a dynamic tissue that comprises the major part of the tooth. Most adhesive procedures in dentistry involve bonding to dentin, to create a strong bond between resin and dentin adequate hybrid Layer formation is essential this bond between dentin and resin adhesive system enhances good marginal adaptation, thereby preventing recurrent caries, microleakage, and pulpal irritation.

To withstand stress generated by Polymerization shrinkage in composite material shear bond strengths of 17–21 MPa value is required. Hence, dental adhesive systems are used to promote adhesion between dental structure and composite resin.[4]
The present study compared shear bond strength of sixth-generation APLP (3M ESPE) and X III (Dentsply) and seventh-generation AEO (3M ESPE) and X IV (Dentsply) adhesives to dentin.

The newer self-etching adhesives are more advantageous as these system include infiltration of the bonding agent into the demineralized dentin and a decreasing the number of clinical procedural steps. The interaction depth of self-etch adhesives at dentin differs from a few hundreds of nanometers depending on the pH of the self-etch solutions. As self-etch adhesives are less acidic, hence they demineralize dentin more superficially than total-etch adhesives. The pH of self-etch adhesives is higher than that of phosphoric acid.

Depending on etching aggressiveness, self-etch adhesives are also classified into three categories based on their pH value: mild (pH of 2.5 or more) demineralize dentin superficially at a depth of 1.0 micron meter and create a thinner transitional layer, moderate (pH of approximately 1), and strong (pH < 1) dissolve the smear layer completely and form a relatively thick transitional layer. In the tested adhesive systems. ADPER EASY ONE (pH=2.3, GROUP C Seventh Generation) showed higher bond strength followed by XENO IV(pH = 2.1, GROUP D), XENO III (pH = 1.5, GROUP B) on dentinal surface, where as ADPER PROMPT L POP (pH =0.7 to 1 Sixth Generation, GROUP A) showed lower bond strength. ADPER APLP (Group A) and X III (Group B) have pH values of less than 1 and 1.4, respectively, and thus are characterized as “strong self-etch” and “intermediary strong self-etch” adhesives. Low bond strength values are present with low-pH self-etching adhesives to dentin due to their initial high acidity that causes deep demineralization.
Jamadar, et al.: An In vitro comparative evaluation of shearbond strength of 6th and 7th generation bonding agents with varying pH.

AEO (Group C) and X IV (Group D) the pH is much higher; hence, they are mild self-etching adhesive; they demineralize dentin upto a depth of 1 µm. They preserve residual hydroxyapatite attached to collagen, and sufficient surface porosity is achieved for micromechanical interlocking through hybridization.\[8\]

The hybrid layer thickness is much smaller in mild self-etching adhesive than strong self-etch or etch-and-rinse approach, but thickness is of minor importance with regard to bonding effectiveness.\[9\]

Additional chemical bonding is achieved by the preservation of hydroxyapatite within the submicron hybrid layer. Higher bond strength is found to have mild self-etching adhesives.

This might be a probable explanation for low bond strength of APLP and X III compared to AEO and X IV.

APLP is a self-etch adhesive composed of Liquid 1 (red blister): methacrylate phosphoric acid, bis-GMA initiators, stabilizers Liquid 2 (yellow blister): water, 2-hydroxyethyl methacrylate (HEMA) polyalkenoic acid, stabilizers.\[10,11\]

However, residual water that remains within the adhesive interface is of a concern because it is hardly removed.

APLP has only water as a solvent, and residual water can be a reason for its low bond strength causing polymerization inhibition, phase separation, and reduced shelf life.

Furthermore, water is a poor solvent for organic compounds such as monomers.\[12,13\]

APLP have (pH = 1) and is considered as strong self-etch adhesive, have shown low bond strength value; this might be because of the absence of solvent. This can be overcome by addition of ethanol a secondary solvent, which causes displacement of residual water and carries the polymerizable monomers into the opened dentin tubules. AEO, X III, and X IV have additional solvent ethanol in it.\[14-16\]

X III, a one-step self-etch adhesive composed of HEMA, ethanol, 2,6-di-tert-butyl-p-hydroxytoluene, nanofiller, urethane dimethacrylate (UDMA) Bisphenol-A-glycidyl dimethacrylate (BHT) camphorquinone, ethyl benzoate, and two new pyro-EMA and PEM-F demonstrated fairly bond strength values with dentin.\[10,17\]

Van Meerbeek et al. credited the good bond strength values with X III because it is an intermediate strong self-etch adhesive, with an acidic pH of 1.4, and this property results in better micromechanical interlocking to enamel and dentin in comparison to mild self-etch adhesives.\[18\]

Furthermore, X III has a high viscosity, which can further decrease bond strength. Insufficient primer drying time reduces bond strength for self-etch adhesives, especially water-based system. Two-step self-etch adhesive systems have been reported to yield higher bond strengths compared to one-step self-etch adhesive systems, due to varying measurement of their chemical constituents. Both contain cross-linking monomers, functional monomers, solvent, inhibitors, and activators but in varying proportions.\[19\]

Less cross-linking monomers were seen with one-step self-etch adhesive systems. These cross-linking monomers provide more mechanical strength.\[20-22\]

The result of the present study revealed that there was a significant difference in the in vitro dentin shear bond strength among the self-etching adhesives tested.
CONCLUSION

The pH values did not influence the shear bond strength significantly in the tested adhesive system. AEO (pH = 2.3, Group C seventh generation) showed higher bond strength than all the tested groups.

RESULTS

In the present study, pH value did not influence the shear bond strength significantly in the tested adhesive systems. AEO (pH = 2.3, Group C seventh generation) showed higher bond strength.

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Conflicts of interest
There are no conflicts of interest.

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