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Original Article

Analysis of micro-shear bond strength of self-etch adhesive systems with dentine: An in vitro study

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ABSTRACT

Background: Success or failure of a composite restoration largely depends on its bonding to enamel/dentine. Several better adhesive systems have been developed during the last few years due to rapid advancement in the technology. Recent self-etched adhesives have fewer clinical steps and are less technique sensitive.

Methods: Ninety extracted human permanent molars were collected, grounded and finished to prepare flat dentine-bonding surfaces on their occlusal surface. All specimens were divided into three groups ($n = 30$) on the basis of three adhesive systems Adper Easy Bond (AE), Beautibond (BB) and Xeno IV (XE). These adhesive systems were applied on prepared mid-dentine-bonding surface. A restorative resin was added with the help of a transparent tube of 2 mm height and 1.7 mm internal diameter and cured. Fifteen specimens in each group were loaded to failure in an Instron Universal Testing Machine after storage for 24 h at 37 °C to check micro-shear bond strength. Another fifteen specimens from each group were thermocycled 500 times at 5 °C and 55 °C with dwell time of 1 min in each bath followed by loading to failure. The data obtained was analyzed with SPSS version 21 at significance level of <0.05 .

Results: After 24 h, micro-shear bond strength of BB was higher (26.04 MPa) than XE (23.69 MPa) and AE (21.50 MPa). After thermocycling, micro-shear bond strength decreased significantly in BB ($P = .001$) and XE ($P = .03$).

Conclusion: The micro-shear bond strength of BB was highest among three groups, which decreased after thermocycling.

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1. Introduction

One of the important factors responsible for the success or failure of a composite restoration is bonding of the material to enamel/dentine. As compared to dentine, enamel has higher mineral content, which helps to bond with composite material satisfactorily. Morphological and structural variations in the structure of dentine present a challenge for the successful bonding. Adhesion mechanism of composite resins to enamel and dentine is based on exchange process, in which resin monomer penetrates into the etched surface of enamel and dentine, which on polymerization micro-mechanically bond to the surface resin tags.^{1,2} However, in this process, the main hindrance is the heterogeneous nature of dentine, with hydroxyapatite deposited on a mesh of collagen fibres.² These minerals are replaced by resin monomers after demineralization of dentine and form an interlocking microstructure known as hybrid layer.³ In addition, dentine contains dentinal tubules with fluid, which traverse from the pulp through the dentine terminating into the dentino-enamel junction.⁴

Rapid advancement in the technology led to development of many better adhesive systems during the last few years. The recent resin-based adhesives are self-etched with fewer steps, simple clinical application and reduced technique sensitivity. In self-etching systems, the separate etching step that is used in the conventional adhesives (etch-and-rinse) is no more required.⁵ Acidic monomers in self-etch systems simultaneously etch and prime the dental hard tissues. Due to their acidic contents, self-etch adhesives are able to remove the smear layer and demineralise the surface of enamel/dentine.⁶ This simplified approach can provide clinically more reliable performance. Although some of these systems have performed satisfactorily both in vitro and in vivo, it seems that the durability of different bonding systems depends on the individual properties of the materials.⁷ Nowadays, concerns have been raised about the bonding effectiveness of different self-etch systems related to their durability. Ageing and adhesive systems both have a significant effect on collagen degradation especially during the first month.⁸

Thus, the present in vitro study was conducted to evaluate the bond strength of some recent self-etch adhesive systems, and also investigate the effect of thermocycling on micro-shear bond strength with dentine.

2. Materials and methods

Ninety freshly extracted human first molars were collected and their root surfaces were cleaned. These teeth were mounted in self-cure acrylic resin blocks and were rinsed with distilled water. A flat dentinal surface of these teeth was prepared by removing 1.5–2.0 mm of their occlusal surfaces with the help of single-sided diamond disc for testing. The superficial dentine surface of each tooth was finished with wet 600-grit silicon carbide paper under running water to produce a standardized smear layer. Following this, these samples were randomly divided into three groups of 30 samples each: Adper Easy Bond (AE) (3M ESPE/ESPE, St. Paul, Minnesota, USA),

Beautibond (BB) (Shofu Dental Corporation, Japan), and Xeno IV (XE) (Dentsply DeTrey, GmbH, Germany).

All the bonding agents were applied according to manufacturer's instruction. A restorative composite resin Tetric N-Ceram (Ivoclar Vivadent AG, FL-9494 Schaan/Liechtenstein) was cured on mid-dentine surface of each tooth with the help of a transparent tube of 2 mm height and 1.7 mm internal diameter. QTH light curing unit XL 2500 (3M ESPE, St. Paul, MN) with visible blue light of 400–500 nm wavelength and radiant exposure of 935 mW/cm² was used for curing. After curing from upside, the tube was removed with the help of number 15 scalpel blade and additional side curing was done in each sample.

These samples were stored in physiologic saline at 37 °C for 24 h. Half of these samples of each group ($n = 15$) were loaded till failure in a Universal testing machine (Unitek, 9450 PC, FIE, India) with a crosshead speed of 1 mm/min. Other half of the samples ($n = 15$) was thermocycled for 500 cycles between water baths at 5 °C and 55 °C with the mean dwell time of 1 min in each bath. These specimens were loaded till failure in Universal testing machine as mentioned above.

3. Statistical analysis

Data were condensed as mean $\pm$ SD. Groups were compared by one-way analysis of variance (ANOVA) and the significance of the mean difference between the groups was done by the Bonferroni post hoc test after ascertaining the normality by Shapiro–Wilk test and homogeneity of variances by the Levene's test. The changes within groups were compared by paired t-test. The Statistical Package of Social Sciences (SPSS) version 21 was used at significance level of $<.05$.

4. Results

The micro-shear bond strength of three adhesive system groups over 24 h and after thermocycling is summarized (Table 1). After 24 h, the mean micro-shear bond strength of BB was the highest, followed by XE and AE with a statistically significant difference ($P = .002$). No significant difference in micro-shear bond strength among the groups was found after thermocycling. The mean micro-shear bond strength of all three bonded groups decreased after thermocycling. However, the decrease was significant in BB ($P = .001$) and XE ($P = .03$). The decrease was higher in BB (17.7%) followed by XE (11.3%) and AE (3.9%) (Table 1).

Table 1 – Shear bond strength (MPa) of three bonded adhesive systems at 24 h and after thermocycling.

Groups	After 24 h	After thermocycling	P-value
AE	21.50 $\pm$ 7.68	20.66 $\pm$ 7.82	.34
BB	26.04 $\pm$ 7.49	21.43 $\pm$ 7.69	.001
XE	23.69 $\pm$ 8.51	21.01 $\pm$ 8.64	.03
P-value	.002	.89	

Table 2 – Composition and manipulation techniques of three adhesive systems used in the study.

Adhesive system	Manufacturer	Composition	Mode of application
Xeno IV	Dentsply, Milford, DE	Adhesive: urethane dimethacrylate resin, hydroxyethylmethacrylate resin, Diethyleneglycol dimethacrylate resin, dihydroxypropane methacrylic and acrylic acid ester resin, dipentaerythritol pentacrylate phosphate, ammonium fluoride salt, photoinitiators photoaccelerators and crosslinker stabilizer Activator: acetone, urethane dimethacrylate resin, hydroxyethylmethacrylate resin, arylborate salt, photoinitiators, stabilizer	Scrub two consecutive coats of adhesive with disposable applicator for 15 s each Air-thin adhesive with mild stream of air for 5 s Light-activate adhesive for 10 s
Adper Easy Bond	3M ESPE, St. Paul, MN, USA	Methacrylated phosphoric esters, dimethacrylates, 2-hydroxyethyl methacrylate, polyalkenoid acid copolymer, colloidal silica, ethanol, water and photoinitiator	Apply bonding agent with gentle agitation for 20 s. Dry for 5 s. Light-cure for 10 s
BeautiBond	Shofu, Kyoto, Japan	4-Methacryloyloxyethyl trimellitic acid, 2,2-bis [4-(2-hydroxy-3-methacryloyloxypropyl) phenyl]propane, Triethyleneglycol dimethacrylate, 6-MHPAc, Acetone, Water and others	Apply BeautiBond and leave for 10 s. Gently air dry for approximately 3 s and then blow with more force. Light cure with a halogen (10 s) or LED (5 s) curing light.

5. Discussion

Long-term success of the composite resin depends on bonding with tooth surface through bonding agent. In this study, micro-shear bond strength of BB was slightly higher than both AE and XE. The result may be due to difference in chemical composition of adhesive resin systems. BB contains 6-methacryloyloxyhexyl phosphonoacetate (6-MHPA), a dual adhesive monomer that claims to have equal bonding efficiency to enamel and dentine, while other systems contain phosphoric acid esters only (Table 2).⁹ After thermocycling, micro-shear bond strength of all three bonded resins decreased. However, the decrease was significant in BB and XE.

Thermocycling is an in vitro process, in which the samples are subjected to temperature extremes compatibilities mimicking the oral cavity environment. This established relationship of linear coefficient of thermal expansion between tooth and the restorative material. Thermocycling stresses are likely to affect the bond strength.¹⁰ The effect of thermocycling on micro-shear bond strength of some adhesive systems to the dentine showed significant reduction in bond strength.¹¹ Price et al. also reported that thermocycling had a very significant negative effect on bond strength with human dentine.¹² This study also showed a significant reduction in micro-shear bond strength in BB and XE. This reduction is possibly due to hydrolysis at the bonded interface and different chemical composition and water sensitivity. Temperature changes and exposure of water are two main factors in thermocycling that influence micro-shear bond strength. The generation of thermal stress due to different coefficient of thermal expansion of bonded substrate (dentine) and material (adhesive systems) may result in bond failure.¹³ Changing the gap dimensions between bonded interface also allows microleakage.^{14,15}

The self-etch systems do not require separate etching and priming steps unlike total etch systems¹⁶; another concern with the self-etch systems is the presence of water or solvent

and it also allows water diffusion, which affects the integrity of restorative interface.¹⁷ Some self-etch adhesives have limited diffusion through smear layer due to their weak acidity. Tooth demineralization with self-etch adhesive has contrary, with tooth buffering capacity and there adhesion could show different behaviour.¹⁸ Acidic solvent of self-etch systems must be neutralized by the mineral content of the tooth structure to facilitate complete polymerization of the adhesive film. The hybridization of resin monomer and demineralised dentinal surface takes place with the self-etch adhesive systems, but at the same time less pronounced resin tag formation is also seen.¹⁹ In total-etch adhesives, the step of rinsing removes all the smear layer and dissolves the mineral. Residual acidity and the presence of smear layer are two main factors that affect the long-term hydrolytic stability of self-etch systems.²⁰ Instead of all, these research showed that self-etch systems produce a better bond strength compare to conventional total-etch systems.²¹

6. Limitations

The limitation of this study was in vitro design causing collagen fibre meshwork to collapse due to dehydration in extracted teeth, which may lead to insufficient bonding. Other factors like pulpal pressure, fluid flow across the dentinal tubule and oral environment may have significant effect on bonding, which cannot be assessed in an in vitro study. Furthermore, long-term ageing also requires evaluation of its effect in establishing a long-term success of composite restorations.

7. Conclusions

The micro-shear bond strength of BB was found to be slightly higher than AE and XE. After thermocycling, micro-shear bond strength of all three bonded adhesive resins decreased.

Conflicts of interest

The authors have none to declare.

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