

## Effect of Thermocycling on Microleakage of New Adhesive Systems on Primary Teeth: An *In-Vitro* Study

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*Received 16 June 2013 and Accepted 13 September 2013*

### Abstract

**Introduction:** This study investigated the sealing ability of three different adhesives in primary bovine teeth. **Methods:** Facial and lingual class V cavities were prepared half in enamel and half in cementum, in 48 bovine primary mandibular incisors and randomly divided into three groups and each group divided to two subgroups. The tested adhesives were XPBond (XP), ClearfilS<sub>3</sub> Bond (S<sub>3</sub>), and Xeno III (XE). All cavities were restored with composite and light cured. After 24 hours storage in 37°C distilled water and polishing, teeth were thermocycled and sealed with nail varnish. Then, they were stored in 2% methylene blue and dye penetration was evaluated under a stereomicroscope. **Results:** No significant differences were recorded in the microleakage value between three adhesives in enamel and dentin margins ( $p>0.05$ ) before and after thermocycling. The lowest microleakage value was obtained in XE followed by XP and S<sub>3</sub>. **Conclusion:** There were not any differences between adhesives in enamel and dentin margins of class V cavities on primary bovine teeth.

**Key words:** Adhesive system, microleakage, primary teeth, thermocycling.

### Introduction

In daily clinical dentistry, there is an increasing demand for aesthetic restorations that has generated intensive research in adhesive materials (1).

The development of adhesive systems is keeping to evolve different versions are constantly being introduced, claiming advantage over their predecessors. Adhesive systems are currently available as etch-and-rinse (three-steps or two-steps) and self-etch (two-step and one-step) and different trade marks are constantly being introduced (2).

Etch-and-rinse adhesives are considered as being complicated and time consuming (3) and tend to be replaced by self-etch adhesives. These systems were reported to reduce the incidence of post-operative sensitivity (4). The development of adhesive systems continues to evolve until the introduction of a single bottle combining etchant, primer and adhesive thus eliminating the additional mixing and/or placement step over the seventh-generation systems in late 2002 (3,5).

Adhesion to enamel is a relatively simple process because enamel is hypermineralized; adhesion to dentin; however, is more difficult owing to higher amount of organic materials and water that can disrupt bonding quality (6). Thermocycling test is the process of subjecting specimens to extreme temperatures in order to simulate changing intraoral temperature conditions (7). It is reported that the effect of thermocycling induce degradation of the tooth/restoration interface due to difference in their coefficient of thermal expansion (8).

The aim of this in-vitro investigation was to evaluate and compare the microleakage in enamel and dentin

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Atash R, Shayegan A, Poureslami H, Sharifi H, Shadman N. Effect of Thermocycling on Microleakage of New Adhesive Systems on Primary Teeth: An *In-Vitro* Study. J Dent Mater Tech 2013; 2(4): 109-13.

margins of class V composite restorations in primary bovine incisors using three adhesives one etch-and-rinse and two self-etch. The null hypothesis was that there were not any significant differences in sealing ability of these adhesives in enamel and dentin margins with and without thermocycling.

### Materials and Methods

Forty eight caries free bovine primary incisors were selected and stored in an aqueous 1% chloramine solution at room temperature. In each tooth, two standardized class V cavities were prepared at the cemento-enamel junction (2 mm above and 2mm below the cemento-enamel junction) on the buccal and lingual surfaces of them, with diamond bur (CF 980204/035 Komet, Lemgo, Germany) of a high-speed handpiece and water coolant spray. Cavity dimensions were 1.5 mm depth, 3mm width and 4mm height. Dimensions were standardized with a periodontal probe. To test three adhesive systems, the specimens were randomly divided into three groups of 16 teeth. Table 1 shows the composition and manufacturers of the tested adhesives.

All materials were applied according to the manufacturer's instruction and all cavities were restored with composite (Grandio, Voco, GmbH, Cuxhaven, Germany) in three increments and each layer was light cured for 40 seconds with a quartz-tungsten-halogen (QTH) light curing unit with 650 mW/cm<sup>2</sup> intensity (Optilux 501, Demetron Kerr, Danbury, CT, USA).

The samples were stored for 24 hours in a 37°C distilled water and restorations were finished with Knife-edge finishing bur and polished with disks then thermocycled in water bath (2500 cycles between 5°C and 55°C, with a dwell time of 15 seconds and a 15 seconds transfer time between baths) to simulate temperature fluctuations in the oral cavity. The teeth for each group were randomly divided into two subgroups (with and without thermocycling).

After thermocycling, the root apices were sealed with sticky wax and covered with two coats of nail varnish up to approximately 1mm of the restoration margins and immersed in 2% methylene blue dye for 24 hours at 37°C.

**Table 1.** Components, the manufacturers and the method of applying of the materials used in this study

Groups	Material	Composition	Manufacturer	Method of applying
XE	Xeno III	<b>Liquid A:</b> -2 hydroxyl ethyl methacrylate (HEMA) -Purified water -Ethanol -Butylated hydroxyl toluene (BHT) -Highly dispersed silicon dioxide <b>Liquid B:</b> -Phosphoric acid modified methacrylate resins -Mono fluorophosphazene modified polymethacrylate resin -Urethane dimethacrylate -Butylated hydroxyl toluene (BHT) -Camphorquinone -Ethyl-4-dimethylaminobenzoate	Dentsply, Detrey, Konstanz, Germany	mixing A + B: 5 seconds application time: 20 seconds Curing time: 10 seconds
S <sub>3</sub>	Clearfil S <sub>3</sub> Bond	MDP, HEMA, bis-GMA, ethanol, initiator, sabilizer, filler	Kuraray dental, Osaka, Japan	application:time: 10 seconds curing time: 10 seconds
XP	XP Bond	PENTA, TCB, UDMA, TGDMA, HEMA, Nanofiller, Camphorquinone, Stabilizer, Tert-Butanol	Dentsply, Detrey, Konstanz, Germany	etching: 15 seconds for dentin, and 30 seconds for enamel application : 20 seconds curing time: 10 seconds

After removal from the dye solution, samples were cleaned, rinsed with tap water, and embedded in slow-curing epoxy resin Epofix (EMS; Fort Washington, PA, USA). After embedding, samples were sectioned labiolingually through the middle of the restoration using a water-cooled diamond disc (Leica 1600 Bensheim, Germany), then abraded with 400 and 600 grit Met Sic paper.

Samples were then examined under a stereomicroscope (magnification: 40X) to determine dye penetration at the enamel and dentin margins of each restoration which were evaluated, separately (Catima Program, Delta logic, Automatisierungstechnik, GmbH, Schwabich, Germany). The microleakage degree was evaluated and scored as follows (9).

0 = no dye penetration.

1 = dye penetration along the incisal or gingival wall less than the total length of the wall.

2 = dye penetration along the entire length of the incisal or gingival wall.

3 = dye penetration along the entire length of the incisal or gingival wall as well as the axial wall.

All the procedures were performed by the same investigator.

For comparison between different adhesive systems, a Kruskal-Wallis test was used. For enamel and dentin margins and for the effect of thermocycling, a Mann-Whitney test was used. Data analysis was done using Stata 11.2 software. Results were considered statistically significant for  $p < 0.05$ .

## Results

None of the adhesives that were used in this study completely prevent microleakage in enamel and dentin margins. Results of microleakages are summarised in Table 2. No significant differences were recorded in the microleakage value between the three adhesive systems on enamel and dentin margins ( $p > 0.05$ ) with and without thermocycling. The best seals in enamel and

dentin margins were obtained in XE followed by XP and S<sub>3</sub>.

## Discussion

Microleakage at teeth-restoration interface is considered to be a major factor influencing the longevity of a dental restoration. It may lead to marginal discoloration and secondary caries (10,11).

In the current study, the null hypothesis was confirmed for all three adhesives that there were not any significant differences in sealing ability of them in enamel and dentin margins with and without thermocycling.

A clinical trial is the most effective method to assess the quality of the bonding systems however the continuous and fast progress of adhesive restorative materials combined with high costs and the immediate demand for information, does not allow for long-term clinical trials (12).

In this study, comparison the results of dentin microleakage showed no significant difference among groups. Excessive etching of the dentin, air drying after etching and amount of moisture have been shown to be critical when using etch-and-rinse adhesive systems (2,13). But in self-etch adhesives, etching and resin infiltration are occurred simultaneously and therefore they are not technique sensitive such as etch-and-rinse systems. Recent studies have suggested that combining the primer and adhesive resins into a simple application step may reduce hybridisation effectiveness (2,14). Some studies concluded that bonding quality in etch-and-rinse and self-etch adhesives were similar (15-17). This result was in agreement with current study. The presence of water in XE is an advantage because water rehydrates dentin and it helps good penetration of collagen network. Self-etch and etch &rinse adhesive systems both showed higher leakage at the dentin margins; however no significant difference was revealed between the individual adhesive systems.

**Table 2.** Microleakage scores on enamel and dentin margins, with and without thermocycling in three adhesives

Margin	Microleakage score	Without thermocycling			With thermocycling		
		XE	XP	S <sub>3</sub>	XE	XP	S <sub>3</sub>
Enamel	0	15	14	13	15	13	12
	1	1	2	2	1	2	3
	2	0	0	1	0	1	1
	3	0	0	0	0	0	0
Dentin	0	15	13	13	12	10	9
	1	1	2	3	2	4	4
	2	0	1	0	2	2	2
	3	0	0	0	0	0	1

XE: Xenon III, XP: XP Bond, S<sub>3</sub>: Clearfil S<sub>3</sub> Bond

These results were in accordance with some studies, however other studies showed a significant difference between self-etch and etch-and-rinse adhesives at the dentin margins (3,18) and there were shown that microleakage in enamel was lesser than dentin margins because of their structural differences (19,20).

Problems related to etching efficacy of enamel in self-etch adhesives, are more common in ones with mild to moderate pH (21).

The results obtained in this study showed that XE provide the best seal followed by XP and S<sub>3</sub> bond but no significant differences were recorded with microleakage degree between the three adhesives on enamel and on dentin before and after thermocycling .

This result also can be explained by a combination of different factors including influence of the pH value, influence of the solvent, and influence of filled/unfilled adhesives.

XE, S<sub>3</sub> and XP are effectively considered to be filled adhesives (22). Because the adhesive layer obtained with these adhesives was thicker, the ability of the interfaces to maintain adhesion during the critical early stages of polymerisation was better, improving the resistance to dimensional changes (23). The presence of HEMA in XE, S<sub>3</sub> ingredients, prevents phase separation and collagen network collapse and helps better adhesive diffusion (24).

S<sub>3</sub> demineralises dentin only to a depth of 1 µm. Moreover, this superficial demineralisation occurred only partially, keeping residual hydroxyapatite still attached to the collagen. Nevertheless, sufficient surface porosity was created to obtain micromechanical interlocking through hybridisation. The preservation of hydroxyapatite within the submicron hybrid layer may serve as a receptor for additional chemical bonding. Furthermore, S<sub>3</sub> contains MDP (methacryloxy-decyl-dihydrogen phosphate), which has a chemical bonding potential to the calcium in the residual hydroxyapatite (25). The hydroxyapatite crystals that remain around the collagen are considered particularly advantageous. Enabling more intimate chemical interaction with the functional monomers on a molecular level, they may also help prevent or retard marginal leakage (26).

For XP's procedure, phosphoric acid is first used to demineralize the dentin. This means that nearly all hydroxyapatite is removed from collagen and thus any chemical interaction between hydroxyapatite and functional monomers is excluded (2).

The self-etch adhesive (XE, S<sub>3</sub>) remain less microleakage value, similar to etch-and-rinse one bottle system (XP) this result is in accordance with the other studies (1,27). The results of this study suggested that the use of self-etch systems with chemical bonding characteristics (such as S<sub>3</sub>) and also lesser clinical steps would be preferable.

## Conclusion

All adhesive system exhibited microleakage at both the enamel and dentin margins. No significant differences were recorded in the microleakage degree between three adhesive systems on enamel and dentin margins before and after thermocycling.

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