Sealing ability and bond strength of four contemporary adhesives to enamel and to dentine

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Abstract

Aim To compare the shear bond strength and microleakage of four adhesive systems to the enamel and dentine of primary bovine teeth. Methods 120 bovine primary mandibular incisors were collected and stored in an aqueous 1% chloramine solution at room temperature for no longer than 3 months after extraction (80 for shear bond testing and 40 for microleakage evaluation). The adhesives tested were Clearfil® SE bond (SE), Adper Prompt L Pop® (LP), Xeno III® (XE), and Prime & Bond NT® (PB). For shear bond strength testing the specimens were wet ground to 600 grit SiC paper to expose a flat enamel or dentine surface. After bonding and restoration with Dyract AP® (DAP), the teeth were subjected to shear stress using a universal testing machine. For microleakage evaluation, facial class V cavities were prepared half in enamel and half in cementum. All cavities were restored with DAP. After thermocycling and immersion in 2% methylene blue, the dye penetration was evaluated under a stereomicroscope. Statistics All data were analysed by Chi-square tests or Fisher’s tests when adapted in order to determine the significant differences between groups. Results were considered as significant for p < 0.05. Results were analysed with an ANOVA test and a Bonferroni’s multiple comparison. The level of significance was p < 0.05. Results Shear bond strength values (MPa) ranged from: on enamel 11.06 to 5.34, in decreasing order SE, LP, XE and PB and on dentine 10.47 to 4.74, in decreasing order SE, XE, LP and PB. Differences in bond strengths between the four systems on enamel and dentine were all statistically significant, excepted for XE vs LP (shear bond at dentine). No significant differences were recorded in the microleakage degree between the four adhesive systems on enamel and on dentine (p >0.0.5). Conclusions The highest shear bond strength was achieved by Clearfil SE bond and the lowest by Prime & Bond NT. There was no significant difference concerning the sealing ability of the four adhesive systems.

Keywords: Microleakage, Shear bond, Self-etching adhesive, Compomers, Primary bovine teeth.

Introduction

The durability of restorative materials is usually measured using dental amalgam as a standard and, subsequently, composite resins and especially conventional glass ionomer cements are reported with a higher failure rate [Kilpatrick, 1993]; therefore the search for improvements of the mechanical properties of glass ionomer cements led to the incorporation of light cured resin components resulting in the introduction of compomers. The latter, or polyacid-modified resin based composite resins (CR), were developed in the 1990’s and combine the technology of glass-ionomer cements (GIC) and resin-based composites with adequate handling characteristics and colour matching [Somphone et al., 2002]. Moreover the physical properties of compomers are similar to those of resin-based composites [Attin, 1996]. Compomers contain fluorosilicic acid glass and polymerisable acidified monomers and have been reported to release fluoride after water absorption from the oral environment [El Kalla and Garcia-Godoy, 1999]. Because of their high resin content, compomers can be bonded to dentine with dentine bonding agent similar to those used for bonding resin composites to dentine, even though specific bonding systems were developed by some manufacturers. However, bond strengths of bonding systems specific to compomers have been reported to be inferior to those of dentine bonding systems for resin-based composite [Wild et al., 1998] at least on permanent teeth. A contemporary approach to dentine and enamel adhesion involves the use of acidic or self-etching...
primers, which combine acid conditioning with the priming procedure, and self-etching adhesive systems were thus developed to simplify the bonding process. Based on the use of non-rinsed acidic polymerisable monomers which serve as conditioner, primer and bonding resin they form a continuous layer between the composite resin and the tooth surface, which is simultaneously demineralised with acidic monomers and penetrated by bonding agents [Perdigão et al., 1997]. The adhesion of materials to dental structures is a relevant issue in dentistry, and the constant and rapid introduction of new adhesive agents can be a problem for the evaluation and clinical selection of a specific product. In order to evaluate the bond strength of an adhesive, shear bond strength has been reported to be the most clinically relevant parameter [Naguchi H et al., 1982].

Microleakage, leading to marginal discolouration and subsequent recurrent carious decay, is also clinically relevant to evaluate a possible failure of an adhesive restoration. As it is difficult to obtain large numbers of intact extracted human teeth for conducting bond strength tests, especially for studies on primary dental tissues, bovine teeth can be considered as possible substitutes for human teeth in adhesion tests [Vanden Abbeele et al., 2004]; primary bovine teeth were used herein. The purpose of this study was, therefore, to evaluate and compare the shear bond strength and microleakage of a self-priming dentine bonding agent (Prime & Bond NT®) to three self-etching adhesive systems (Adper Prompt L Pop®; Clearfil SE Bond®; Xeno III®).

Materials and methods
A total of 120 non-carious bovine mandibular primary incisors were collected and stored at 4°C in an aqueous 1% chloramine solution for no longer than 3 months after extraction.

Shear bond strength tests. Eighty (80) incisors were used for shear bond determination. The roots of the teeth were removed with a separating disc and the labial surfaces of the primary incisors were used. The detailed preparation of the teeth for these tests has been described previously [Atash and Vanden Abbeele, 2005]. A polytetrafluoroethylene jig was placed over each specimen to limit and standardise the area available for bonding to 2 mm in diameter [Agostini et al., 2001; Atash and Vanden Abbeele, 2005]. For each type of surface, the specimens were randomly divided into four adhesive treatment groups of 10 specimens each.

Shear bond strength. For each group, the specimens were embedded in slow curing bonding procedures. Four commercial adhesive systems – Clearfil SE Bond® (SE) (Kuraray Dental, Osaka, Japan), Adper Prompt L Pop® (LP) (3M Dental Products, St Paul, MN, USA), Xeno III® (XE) (Dentsply, Detrey, Konstanz, Germany), and Prime & Bond NT® (PB) (Dentsply, Detrey, Konstanz, Germany) – were used in this study and applied according to the manufacturers’ instructions. The composition of all the materials used is listed in Table 1. The further surface treatments and use of a polytetrafluoroethylene mould has been described previously [Atash and Vanden Abbeele, 2005].

Microleakage tests. Forty incisors were used for microleakage studies. Ten teeth were randomly selected and used for each tested adhesive system.

Cavity preparation. One standardised C-shape class V cavity was located at the cementum-enamel junction, on the buccal surface of each tooth. Cavities were prepared with diamond burs (CF 980204.035, Komet, Lemo, Germany) on a high-speed hand piece with water cooling. The cavities were located on the cementum-enamel junction, half in enamel and half in cementum. Occlusal (enamel) cavo surface margins were bevelled to approximately 45° and the gingival (cementum) cavo surface margins were left at 90°C. Cavity dimensions were 1.5 mm depth, 3 mm width, and 4 mm height. The length of the bur was used as a guide for cavity depth.

Cavity restoration. Each cavity was cleaned with pumice using a rubber cup prior to restoration. The adhesive systems were applied strictly according to the manufacturers’ instructions and all cavities restored with compomer Dyract AP (Dentsply, Detrey, Konstanz, Germany).

Dye penetration. The samples were first stored 24 hours in a saline solution at 37°C and then thermally cycled in water baths: 2500 cycles between 5 and 55°C with a dwell immersion time of 15 seconds and 15 seconds transfer baths, to simulate temperature fluctuations found in the oral cavity. After thermocycling, the teeth were covered with two coats of nail polish up to approximately 1 mm of the restoration margin and immersed in 2% methylene blue dye for 24 hours at 37°C. After removal from the dye solution the teeth were cleaned, rinsed with tap water, and embedded in slow-curing epoxy resin Epofix (EMS; Fort Washington, PA, USA). After embedding, the teeth were sectioned labiolingually through the middle of the restoration using a water-cooled diamond disc. The different samples were then examined under a stereomicroscope (magnification: 125x) to analyse dye penetration at the marginal seal of each restoration (Catima Program, Deltalogic).
Microleakage degree. This was evaluated and scored as follows.
- 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.

Statistical analysis. For the dentine and enamel groups, all shear and tensile bond strengths values were compared with an ANOVA and Bonferroni’s multiple comparison test.

All data were analysed by Chi-square tests or Fisher’s tests when adapted in order to determine the significant differences between groups. Results were considered as significant for p < 0.05.

Results

Shear bond strength values on enamel decreased from 11.06 for SE, 9.34 for LP, 7.55 for XE and 5.34 for PB. In the tests on dentine, the values decreased from 10.47 for SE, 6.60 for XE, 6.10 for LP and 4.74 for PB.

These results are shown in Tables 2 and 3. Differences were statistically significant for all pairs using a Bonferroni’s test, excepted for XE versus LP on dentine.

Tables 4 and 5 show the microleakage scores for the adhesives on enamel and on cementum. Except for LP, none of the adhesives showed microleakage on enamel (score 0 in the 50 samples for SE, XE and PB, and in 49 samples for LP) (1 sample with score 1). All samples showed some microleakage (score 1) on cementum, ranging from 1 sample with microleakage for XE, 2 for PB, 4 for LP and 5 for SE. However, none of these differences were significant.
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**Discussion**

Mechanical testing of adhesion provides some knowledge on the adhesive properties of bonded materials. Shear stress is considered to be more representative of the clinical situation [Naguchi et al., 1982]. Moreover clinicians and researchers use microleakage as a measure for assessing the performance of restorative materials in the oral environment [Bauer and Henson, 1984]. Dye penetration measured on sections of restored teeth is the most common technique for evaluating microleakage at the tooth-restoration interface [Alani and Toh, 1997].

Because the differences in tubule diameters and the number of lateral branches may have some effect on dentine bond strength, bovine superficial dentine was used as a substitute for human dentine [Ferrari and Davidson, 1996].

Previous studies [Van Meerbeek et al., 2001; Atash and Vanden Abbeele, 2004; Atash et al., 2004] have shown that bond strength and microleakage depend on the several factors, including:

- influence of the pH value;
- influence of the solvent;
- influence of filled/unfilled adhesives.

**Influence of pH.** SE (pH = 2) is considered as a “mild self-etching adhesive” [Van Meyerbeer et al., 2001]. This study found SE to have the highest bond strength, and are in agreement with the findings of several other studies [Atash et al., 2004]. SE demineralises dentine only to a depth of 1 µm, and this superficial demineralisation occurs only partially, keeping residual hydroxyapatite still attached to the collagen. Nevertheless, surface porosity is sufficient to create micromechanical interlocking through hybridisation. This hydroxyapatite, present within the submicron hybrid layer, may provide an additional chemical bonding to MDP (methacryloxydihydrogen phosphate) present in SE [Van Meerbeek et al., 2001].

The hydroxyapatite crystals remaining around the collagen also enable a more intimate chemical interaction with the functional monomers on a molecular level and may help prevent or retard marginal leakage [Van Meerbeek et al., 2003].

The pH of XE is about 1.4, and this adhesive is more acidic than the “mild” self-etching adhesives, providing a better micromechanical interlocking at the enamel and the dentine. The residual hydroxyapatite at the hybrid layer base may still allow some chemical intermolecular interaction, as demonstrated previously for the “mild” self-etching adhesives [Van Meerbeek et al., 2003].

LP (pH=0. 4) [Van Meerbeek et al., 2001] is considered as a “strong self-etching adhesive”. For enamel, the resulting acid-etch pattern resembles a phosphoric-acid treatment following a three-step
adhesive systems approach [Van Meerbeek et al., 2001]. For dentine, the collagen is exposed after dissolving nearly all the hydroxyapatite corresponding to the hybrid layer. Such low-pH self-etching adhesives have been documented with rather low bond strength values, especially to dentine [Inoue et al., 2001], which is confirmed in this study.

The low micro-leakage observed with Adper Prompt L Pop could be attributed to the presence of phosphoric esters in its formulation. Another explanation is that the lower PKa of Adper Prompt L Pop is sufficient to etch beyond the smear layer and demineralise the underlying intact dentine with the formation of an authentic hybrid layer [Gagliardi, 2002].

Influence of the solvent. LP contains water as a solvent. The bonding performance may be affected by the residual solvent, which is difficult to remove completely and thus remains within the adhesive interface [Van Meerbeek et al., 1998]. The high water content (80%) of LP may result in competition between the monomer and the water remaining inside the demineralised dentine [Van Meerbeek et al., 1998]. Phase section of the hydrophobic and hydrophilic monomer components, causing blister-like spaces and globule formation of the resin within the hybrid layer, has been observed in overly wet conditions [Kaaden et al., 2002]. LP is based on water and XE on ethanol. The residual water may interfere with the polymerisation of adhesive monomer, thereby diminishing the quality of the hybrid layer [Gagliardi and Avelar, 2002].

Influence of filled/unfilled adhesives. XE and SE are considered as filled adhesives [Van Meerbeek et al., 1998]. Because the adhesive layer obtained with these adhesives is thicker, the ability of the interfaces to maintain adhesion during the critical early stages of polymerisation is better, improving the resistance to dimensional changes. Studies have shown that the use of low-rigidity resins improved the stain capacity of the restoration and significantly influenced the quality of the marginal integrity [Van Meerbeek et al., 1993].

The progressive loss of marginal integrity and subsequent marginal discoloration by microleakage is probably still caused mainly by residual stresses from polymerisation shrinkage of the composite restorative material and stresses resulting from thermal dimensional changes [Van Meerbeek et al., 2003]. Incorporation of filler particles into the bonding resin may promote the formation of adhesive films with appropriate thicknesses and also reduce the shrinkage of the adhesive, even if the modulus of elasticity and thus the rigidity of the adhesive will be increased [Labella et al., 1999]. The NRC/PBNT system uses 10% maleic acid to condition the dentine surface in combination with a primer using a non-rinse technique where the smear layer is not rinsed off but incorporated into the adhesive layer, according to the manufacturer. However, smear layer remnants could inhibit the penetration of the primer/resin, resulting in lower shear bond strength values. Consequently, PBNT is unable to condition enamel adequately [Breschi et al., 1999].

PBNT also contains dipentaery thritol penta-acrylate monophosphate (PENTA), a molecule of mild acidity [Perdigão et al., 1994]. Because of this acidity, PENTA might behave as a mild conditioning agent. However, on dentine, the ability to remove the smear layer and plugs will be only slightly modified by PENTA [Eick et al., 1995]. The present results confirm the low shear bond strengths obtained with PBNT in other studies both on enamel and on dentine [Perdigão et al., 1998].

The priming acid of NRC contains carboxylic acid groups that are capable of adhering to the calcium ions of the tooth structure. Better adhesion via calcium ion bonding to the enamel than to cementum may be present [Ruyter, 1992] and this may explain the higher microleakage found.

Conclusion
There are differences between shear bond strengths of different adhesive systems used with a compomer on enamel as well as on dentine, while there are no significant differences in the microleakage degree either on enamel or on cementum. As compomers are widely used in paediatric dentistry, all systems can be used if microleakage is considered as the main clinical parameter, but in cases where shear strengths are important (incisal edges, long lasting restorations) dentists should prefer to use an adhesive with a better performance, such as Clearfil SE Bond®.

References


