Effect of bleaching agents on bonding to pulp chamber dentine

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Abstract

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Aim To determine the effect of intracoronal bleaching agents on adhesion of bonding agents to pulp chamber dentine.

Methodology Forty extracted human maxillary anterior teeth were randomly divided into four groups of 10 teeth each. Bleaching agents were sealed in pulp chambers for 7 days, as in clinical use. Group 1 (control): distilled water, group 2: 35% hydrogen peroxide, group 3: sodium perborate mixed with water, and group 4: sodium perborate mixed with 35% hydrogen peroxide. Teeth were stored in saline at 37 °C for 7 days. After the bleaching agent was removed, teeth were leached in water for a further 7 days prior to bonding. The crown was cut vertically from mesial to distal and the labial pulp chamber dentine was prepared for bonding with Clearfil SE-Bond and filled with resin composite (Clearfil AP-X). The bonded specimens were kept moist at 37 °C for 24 h. Microtensile bond strengths were determined using a universal testing machine. Additional teeth were prepared using the same bleaching procedures to investigate the scanning electron microscopic appearance of the dentine surface.

Results Mean values (±SD) of microtensile bond strength for the experimental groups were: group 1: 5.29 ± 2.21 MPa, group 2: 5.99 ± 1.51 MPa, group 3: 9.17 ± 1.65 MPa and group 4: 3.99 ± 1.31 MPa. Dentine treated with sodium perborate in water (group 3) had significantly higher mean bond strength when compared with the other three groups ($P < 0.05$, Tukey's test). Mean bond strength was lowest when dentine was treated with sodium perborate plus hydrogen peroxide (group 4).

Conclusions In terms of subsequent bond strength during restoration, sodium perborate mixed with distilled water appears to be the best intracoronal bleaching agent.

Keywords: bleaching agents, bonding, dentine, pulp chamber.

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Introduction

Root filled teeth may turn dark and lose translucency after loss of vitality. The discolouration of teeth with nonvital pulps requires an effective treatment using chemical bleaching agents. Bleaching of root filled teeth was first reported by Dwinelle (1850). Nutting & Poe (1967) sealed 35% hydrogen peroxide into the coronal pulp chamber and removed it 3 weeks later when the required result had been achieved; they called this technique the 'walking bleach'. A technique using sodium perborate mixed with water was suggested in order to minimize the risk of cervical root resorption (Spasser 1961). Hydrogen peroxide (30%) mixed with sodium perborate was later proposed as a bleaching agent because bleaching efficacy is enhanced (Ho & Goerig 1989). Currently the most commonly used intracoronal bleaching materials are hydrogen peroxide (H_2O_2) and sodium perborate ($NaBO_3 \cdot 4H_2O$) (Rotstein & Walton 2002). Although these agents are effective in lightening tooth colour (Kaneko *et al.*

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2000), their use has been associated with undesirable complications, including increased dentine permeability, changes in tooth structure, external root resorption, microleakage of restorations and reduced bond strength of composite resins (Crim 1992, Stokes *et al.* 1992, Titley *et al.* 1993, Chng *et al.* 2002).

Subsequent to intracoronal bleaching, endodontic access cavities are frequently lined with glass–ionomer cement and restored with bonded composite resin. Ideally, not only the enamel margin but also the entire pulp chamber should be sealed. Unless the glass–ionomer cement liner covers the entire dentine surface, bonding of the resin to dentine as well as enamel is essential to minimize marginal leakage and hence percolation of bacteria and discolouring agents at the cavity margin (Sheets *et al.* 2002). Any change in the surface properties of dentine after bleaching is likely to have an impact on the effectiveness of dentine bonding. Torneck *et al.* (1990) used bovine teeth to evaluate the adhesive properties of composite resin to bleached and unbleached bovine dentine. They reported that bleaching may compromise the adhesive bond strength of the resin, and as such may increase the incidence of restorative failure in complex cavities.

Due to specific properties of dentine, bonding has not yet achieved ideal characteristics (Perdigao *et al.* 2000). Most bonding systems adhere more strongly to superficial dentine, with progressively lower bond strength to deeper dentine (Pashley *et al.* 1993). The dentine walls that make up the pulp chamber are the deepest possible dentine. Tubule diameters are large and tubule density is high, making it a more challenging bonding substrate (Belli *et al.* 2001). Thus, the pulpal surface of dentine has different characteristics for bonding than those of cut dentine that is usually encountered in operative dentistry.

When the adhesive material is used properly, there is usually no gap between these materials and tooth structure, greatly reducing microleakage. Application of adhesives to acid-etched dentine creates an acid-resistant, resin-infiltrated collagen layer, the so-called hybrid layer that not only retains composites to dentine, but also seal dentine against the ingress of oral fluids (Youngson *et al.* 1990, Sano *et al.* 1995). When an access cavity is restored with resin composite, it is important to bond to pulp chamber dentine to enhance the adhesive area and hence the marginal seal.

Many new adhesive systems have been introduced. The main feature has been simplification of the bonding procedures and a decrease in the time needed for application. These adhesive systems include 'single-bottle' systems, which combine priming and bonding into one step, and 'self-etching priming' systems, which combine conditioning and priming into one step (Tanumiharja *et al.* 2000). The self-etching primer system provides a simpler bonding technique, with greater bond strengths to dentine than total etching (Kijsamanmith *et al.* 2002).

Bond strength is significantly reduced when bonding is performed immediately after bleaching. Oxygen produced during bleaching remains in the enamel and dentine for up to 2 weeks, and may interfere with the chemistry of bonding agents (Titley *et al.* 1992, 1993). The purpose of this study was to determine the effect of intracoronal bleaching agents (35% hydrogen peroxide, sodium perborate and sodium perborate mixed with 35% hydrogen peroxide solution) on adhesion of bonding agent to pulp chamber dentine. The null hypothesis tested in this study was that bleaching agents do not reduce bond strength between bonding agent and dentine.

Materials and methods

Selection and preparation of teeth

Forty extracted human maxillary anterior teeth with intact crowns were collected and stored at 4 °C in normal saline solution. The criteria for tooth selection were: complete root formation, no caries, no restoration and no fracture line. The teeth were thoroughly cleaned. During subsequent preparation, care was taken to prevent dehydration of the specimens by keeping all teeth wrapped in gauze moistened with water. A conventional endodontic access cavity was prepared in each tooth, using a diamond bur in a high-speed hand piece under water coolant. The pulp tissue was removed and cleaning and shaping was carried out using the stepback technique. Canals were irrigated with 2 mL 2.5% sodium hypochlorite between successive files. Each canal was irrigated with 5 mL 2.5% sodium hypochlorite as a final rinsing of the canal. Then the root canal was dried with paper points. The coronal part of the root canal was packed with Caviton (GC Caviton; GC Dental Products Corp., Tokyo, Japan) to 4 mm below the cementoenamel junction, using an endodontic plugger.

The teeth were divided randomly into four groups of 10 teeth each, by pooling all prepared teeth and assigning them to the four groups using a random numbers table. Bleaching agents were placed into the pulp chambers as follows:

Group 1: (control) cotton pellet soaked with distilled water.

Group 2: 35% hydrogen peroxide on a cotton pellet filling the pulp chamber (Opalescence Endo; Ultradent Products Inc., South Jordan, UT, USA).

Group 3: sodium perborate (Pharmaceutical Department, Chulalongkorn University, Bangkok, Thailand) mixed with distilled water in a ratio of 2 g powder to 1 mL liquid. A similar quantity (about 0.1 g sodium perborate and 0.05 mL liquid) of bleaching paste was placed into the pulp chamber of each tooth.

Group 4: sodium perborate mixed with 35% hydrogen peroxide (APS Ajax Finechem, Melbourne, Victoria, Australia) in the same ratio as for group 3, and a similar quantity of bleaching paste was placed into the pulp chamber.

Access cavities were sealed with 4 mm thickness of Cavit. Each tooth was placed in a capped plastic tube and stored at 37 °C in 100% humidity for 7 days. Cavit and treatment agents were removed from the access cavity and the pulp chamber was rinsed with 20 mL distilled water. A cotton pellet soaked in distilled water was placed in the pulp chamber and sealed with Cavit. The specimens were stored in 100% humidity at 37 °C for 7 days before bonding and microtensile testing.

Preparation of samples for microtensile testing

The root was removed from the crown approximately 2 mm below the cementoenamel junction using a slow speed diamond saw (Accutom-50; Struers, Copenhagen, Denmark) under copious water spray and then cut vertically from mesial to distal to expose labial pulp chamber dentine. The specimens were cleaned with distilled water to remove debris, then air-dried with a triple syringe. The pulp chamber dentine was bonded with Clearfil SE-Bond (Kuraray, Osaka, Japan) according to the manufacturers' instructions, as follows: the primer was thinly applied with disposable brushes for 20 s, dried with a mild air flow, and the bonding agent was applied, air-flowed gently and light-cured for 10 s. A block of resin composite (Clearfil AP-X; Kuraray, Osaka, Japan) was built up on the bonded surface and light cured. The specimens were then kept in tap water for 24 h at 37 °C.

Each specimen was sectioned in a bucco-lingual direction to provide two sections each 0.7 mm thick (Fig. 1). Thus 20 specimens were prepared from each group. As described in detail previously (Sano *et al.* 1994), these sections were then shaped to a dumbbell shape with the narrowest portion at the bonded interface and standardized to produce a bonded surface area of 1.0 ± 0.2 mm^2 by using a superfine diamond bur (Intenzive; Swiss Dental Products, Zurich, Switzerland) with a high-speed hand piece under copious air–water spray. The thickness and width of the bonded area of each specimen were checked before testing using a digital micrometer (Mitutoyo Corp., Tokyo, Japan). The specimens were then attached to a Bencor-Multi-T testing apparatus (Danville Engineering, Danville, CA, USA) with a cyanoacrylate adhesive (Zap-it; DVA, Corona, CA, USA) and stressed in tension using a universal testing machine (Instron, 5566series5000, London, UK) at a crosshead speed of 1 mm min^{-1}. The mean microtensile bond strengths (MPa) at failure mode were calculated as the maximum load at failure divided by the bonded cross-sectional area.

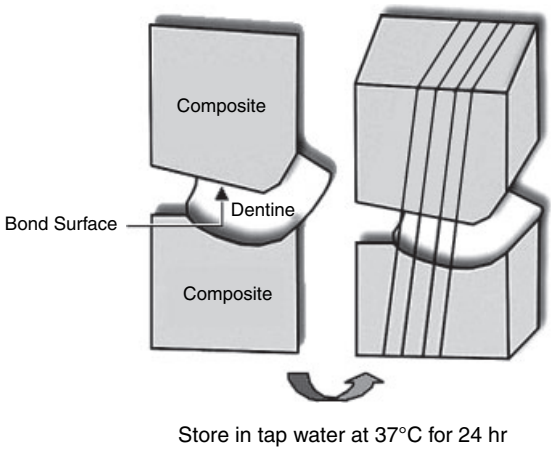

Figure 1 Schematic drawing demonstrated a composite resin was built up on the bond surface of the pulp chamber, and the specimen was serially sections.

Statistical analysis

The mean bond strengths were statistically analysed using multiple comparison range tests, followed by Tukey's test. A P-value <0.05 was considered to be significant.

SEM observation

Failure mode

The fractured surfaces were air-dried, mounted on an aluminium stub and processed for scanning electron microscopy (SEM) (JSM-5410 LV; JEOL, Tokyo, Japan) at 75× magnification to determine the mode of failure after microtensile testing.

Investigation of the dentine surface

The pulpal dentine surface in the region for bonding was observed using SEM.

Fifteen teeth were prepared (three teeth for each of the bleaching procedures plus three specimens with no treatment). After rinsing, the treated and untreated dentine surfaces were air-dried and gold sputter-coated.

Results

The effects of the various bleaching agents on the microtensile bond strength of Clearfil-SE bond to pulp chamber dentine are shown in Table 1. The mean bond strength to dentine irrigated with 2.5% NaOCl during cleaning and shaping but not treated with bleaching agent (control) was 5.29 ± 2.21 MPa (mean ± SD, $n = 20$). Mean microtensile bond strength was highest for dentine treated with sodium perborate in distilled water (9.17 ± 1.65 MPa) and lowest for dentine treated with sodium perborate plus hydrogen peroxide (3.99 ± 1.31 MPa), with an intermediate value for hydrogen peroxide alone. Multiple comparisons showed a highly significant effect of treatment with bleaching agents ($P < 0.01$). The mean bond strength to dentine treated with sodium perborate in water (group 3) was significantly higher than for the other three groups including the control (group 1), and the only other statistically significant difference was between groups 2 and 4 ($P < 0.05$).

SEM observations

The failure mode in all groups was predominantly mixed failure (type 2; partial cohesive failure in dentine or partial cohesive failure in bonding resin/composite). Two specimens from each of groups 2 and 4, both involving exposure to hydrogen peroxide, showed adhesive failure (type 1).

Table 1 Microtensile bond strength to pulp chamber dentine ($n = 20$)

	Mean	SD	SEM	Max	Min
Group 1: control	5.29[a,b]	2.21	0.49	8.96	2.01
Group 2: hydrogen peroxide	5.99[b]	1.51	0.34	9.21	4.22
Group 3: sodium perborate + distilled water	9.17[a]	1.65	0.37	11.91	6.77
Group 4: sodium perborate + hydrogen peroxide	3.99[c]	1.31	0.29	6.18	2.29

Group identified by different superscript letters are significantly different ($P < 0.05$).

Pulp chamber dentine before the bonding procedure typically showed various sizes of dome-shaped calcospherites with open dentinal tubules (Fig. 2). Sodium perborate in distilled water did not visibly alter the dentine surface. When the pulp chamber was treated with hydrogen peroxide (group 2) or sodium perborate mixed with hydrogen peroxide (group 4), the dentine surface was different in appearance. Calcospherites were less pronounced, with a much flatter dentine surface, whilst tubules remained patent (Fig. 3). No differences in the dentine surface were noted between groups 2 and 4.

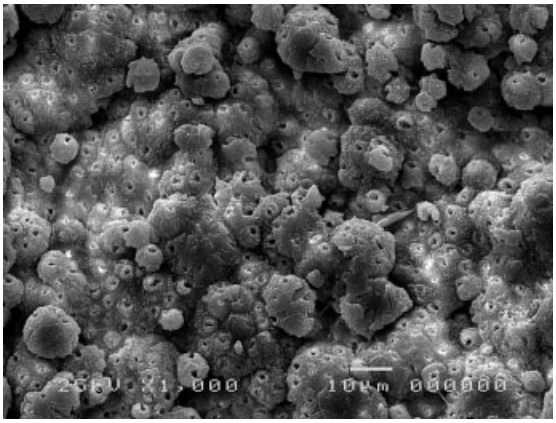

Figure 2 SEM photograph of normal pulp chamber dentine. Various sizes of dome-shaped calcospherites with open dentinal tubules are visible (original magnification, ×1000).

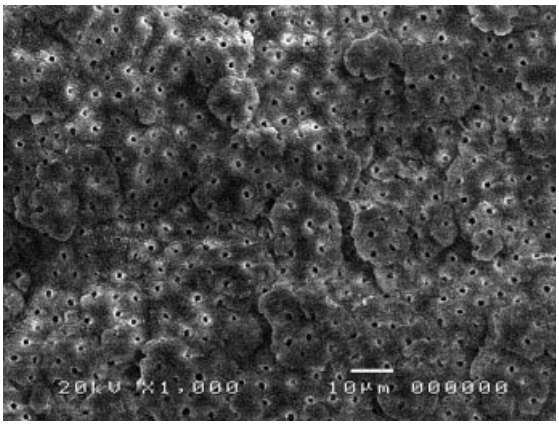

Figure 3 SEM photograph from the group treated with 35% hydrogen peroxide. The surface was altered by the bleaching agent, dentinal tubules were open and partly covered by the smear layer.

Discussions

The microtensile bond strength test used in this study (Sano *et al.* 1994) allows the testing of very small cross-sectional areas of dentine-resin specimens (1.0 ± 0.2 mm^2) and develops a uniform stress distribution during testing. The strength of dental adhesive systems has commonly been evaluated using the shear bond strength test. This traditional method uses large surface areas (about 7–12 mm^2) in testing, which are difficult to achieve in studies of pulpal surface dentine. The typical form of fracture with shear testing (mostly cohesive failure) does not provide reliable information with regard to the adhesive strength of the bond (Erickson *et al.* 1989, Perinka *et al.* 1992). Furthermore, a high bond strength is almost impossible to measure using this method. Later the method of microtensile bond test demonstrated higher bond strengths than other methods that used large surface areas (Sano *et al.* 1994). Since then, a microtensile test method has become commonly used.

Overall, the mean values for bond strength in this study were low because bonding may be affected by differences in the dentine location and changes in the properties in different locations within a tooth. The bond strength to dentine close to the pulp was much less than that to superficial dentine (Suzuki & Finger 1988). Regional variations in permeability of dentine have also been reported (Pashley *et al.* 1987, Maroli *et al.* 1992), related to variations in the density and diameter of dentinal tubules. This study was performed on pulp chamber dentine, which is the deepest possible dentine, with large tubule diameters and high tubule density. In addition, pulpal dentine was exposed to sodium hypochlorite during canal preparation. This is in accordance with previous studies that reported a reduction of bond strength of bovine coronal dentine treated with 5% NaOCl, from 16 MPa to approximately 5 MPa (Nikaido *et al.* 1999, Morris *et al.* 2001).

Several factors may be associated with the adverse effect of hydrogen peroxide on bond strength. Hydrogen peroxide is capable of generating hydroxyl radical, an oxygen-derived free radical which is known to accumulate in dentine and inhibit polymerization of resin (Titley *et al.* 1993). Although this study delayed bonding for 7 days after bleaching, residual oxygen may remain in dentine and retard polymerization. In addition, different properties of bleaching agents, such as the pH of the solutions and decomposition products of each agent may affect the dentine surface. The pH variation amongst bleaching agents was reported by Rotstein & Friedman (1991). Sodium perborate is alkaline, whereas 30% hydrogen peroxide is acidic. The pH of the materials when mixed together gradually changes from acidic to alkaline as the concentration of dissolved sodium perborate gradually increases. A previous study has shown that there was a relation between microhardness and calcium concentration and bond strength (Perinka *et al.* 1992). In this study sodium perborate did not adversely effect bond strength, and actually increased it. This may be related to the absence of a significant reduction in calcium levels or microhardness of dentine following treatment with sodium perborate (Lewinstein *et al.* 1994, Rotstein *et al.* 1996, Chng *et al.* 2002). On the contrary, exposure to high concentrations of hydrogen peroxide decreased dentine microhardness and led to alterations of chemical structure of dentine (Lewinstein *et al.* 1994, Rotstein *et al.* 1996, Teptoranintra *et al.* 2001).

It has been reported that dentine treated with 35% hydrogen peroxide and 35% carbamide peroxide would achieve significantly higher bond strength if bonding treatments were delayed for another week (Spyrides *et al.* 2000). Several studies have suggested that inhibition of resin polymerization by residual oxygen can be reversed by leaching dentine in water for a period of 7 days prior to bonding (Torneck *et al.* 1991, Spyrides *et al.* 2000). This study suggests that high concentrations of hydrogen peroxide should not be used as part of the bleaching process, because of the persistent effect on bond strength even after leaching.

Conclusions

Hydrogen peroxide should be avoided as a bleaching agent for the 'walking bleach' if bonding agents are to be used during subsequent restoration. Pulp chamber dentine bleached with sodium perborate in distilled water exhibited an elevated microtensile bond strength, and it is therefore recommended that, whenever possible, sodium perborate should be used for intracoronal bleaching.

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References

Belli S, Zhang Y, Pereira PNR, Ozer F, Pashley DH (2001) Regional bond strengths of adhesive resins to pulp chamber dentine. *Journal of Endodontics* **27**, 527–32.

Chng HK, Palamara JEA, Messer HH (2002) Effect of hydrogen peroxide and sodium perborate on biomechanical properties of human dentine. *Journal of Endodontics* **28**, 62–7.

Crim GA (1992) Post-operative bleaching: effect on microleakage. *American Journal of Dentistry* **5**, 109–12.

Dwinelle WW (1850) Ninth annual meeting of the American Society of Dental Surgeons Article X. *American Journal of Dental Science* **1**, 57–61.

Erickson R, Glasspoole E, Retief D (1989) Influence of test parameters on dentine bond strength measurements. *Journal of Dental Research* **68**, 374 (Abstract No. 1543).

Ho S, Goerig AC (1989) An in vitro comparison of different bleaching agents in the discolored tooth. *Journal of Endodontics* **15**, 106–11.

Kaneko J, Inoue S, Kawakami S, Sano H (2000) Bleaching effect of sodium percarbonate on discolored pulpless teeth in vitro. *Journal of Endodontics* **26**, 25–8.

Kijsamanmith K, Timpawat S, Harnirattisai C, Messer HH (2002) Micro-tensile bond strengths of bonding agents to pulpal floor dentine. *International Endodontic Journal* **35**, 833–9.

Lewinstein K, Hirschfeld Z, Stabhoz A, Rotstein I (1994) Effect of hydrogen peroxide and sodium perborate on the microhardness of human enamel and dentine. *Journal of Endodontics* **20**, 61–3.

Maroli S, Khera SC, Krell KV (1992) Regional variation in permeability of young dentine. *Operative Dentistry* **17**, 93–100.

Morris MD, Lee KW, Agee KA, Bouillaguet S, Pashley DH (2001) Effects of sodium hypochlorite and RC–Prep on bond strengths of resin cement to endodontic surfaces. *Journal of Endodontics* **27**, 753–7.

Nikaido T, Takano Y, Susafuchi Y, Burrow MF, Tagami J (1999) Bond strengths to endodontically treated teeth. *American Journal of Dentistry* **12**, 177–80.

Nutting EG, Poe GS (1967) Chemical bleaching of discolored endodontically treated teeth. *Dental Clinics of North America* **11**, 655–62.

Pashley DH, Andringa JH, Derkson GD, Derkson ME, Kalathoor SM (1987) Regional variability in the permeability of human dentine. *Archives of Oral Biology* **32**, 519–23.

Pashley EL, Tao L, Matthews WG, Pashley DH (1993) Bond strengths to superficial, intermediate and deep dentine in vivo with four dentine bonding systems. *Dental Materials* **9**, 19–22.

Perdigao J, Frankenberger R, Rosa BT, Breschi L (2000) New trends in dentine/enamel adhesion. *American Journal of Dentistry* **13** (Spec. No.), 25D–30D.

Perinka L, Sano H, Hosada H (1992) Dentine thickness, hardness, and Ca – concentration vs bond strength of dentine adhesives. *Dental Materials* **8**, 229–33.

Rotstein I, Friedman S (1991) pH variation among materials used for intracoronal bleaching. *Journal of Endodontics* **17**, 376–9.

Rotstein I, Walton RE (2002) Bleaching discolored teeth: internal and external. In: Walton R, Torabinejad M, eds. *Principles and Practice of Endodontics*, 3rd edn, Chapter 23, pp. 405–423. Philadelphia: WB Saunders Company.

Rotstein I, Dankner E, Goldman A, Heling I, Stabhoz A, Zalkind M (1996) Histochemical analysis of dental hard tissues following bleaching. *Journal of Endodontics* **22**, 23–6.

Sano H, Shono T, Sonoda H et al. (1994) Relationship between surface area for adhesion and tensile bond strength – evaluation of a microtensile bond test. *Dental Materials* **10**, 236–40.

Sano H, Shono T, Takatsu T, Ciucchi B, Russel CM, Pashley DH (1995) Nanoleakage: Leakage within the hybrid layer. *Operative Dentistry* **20**, 18–25.

Sheets C, Paquette J, Wright R (2002) Tooth-whitening modalities for pulpless and discolored teeth. In: Cohen S, Burns RC, eds. *Pathways of the Pulp*, 8th edn, Chapter 21, pp. 755–757. St Louis, MO: Mosby Inc.

Spasser HF (1961) A simple bleaching technique using sodium perborate. *New York Dental Journal* **27**, 332–4.

Spyrides GM, Perdigao J, Pagani C, Araujo MA, Spyrides SM (2000) Effect of whitening agents on dentine bonding. *Journal of Esthetic Dentistry* **12**, 267–70.

Stokes AN, Hood JAA, Dhariwal D, Patel K (1992) Effect of peroxide bleaches on resin- enamel bonds. *Quintessence International* **23**, 769–71.

Suzuki T, Finger WJ (1988) Dentine adhesives: site of dentine vs bonding of composite resin. *Dental Materials* **4**, 379–83.

Tanumiharja M, Burrow MF, Tyas MJ (2000) Microtensile bond strengths of seven dentine adhesive systems. *Dental Materials* **16**, 180–7.

Teptoranintra S, Benjakul P, Dhanasomboon S, Cheumarrom C (2001) Influences of various bleaching agents on fracture resistance of endodontically treated anterior teeth. *Journal of Dental Research* **80**, 1378.

Titley KC, Torneck CD, Ruse ND (1992) The effect of carbamide peroxide gel on the shear bond strength of a microfil resin to bovine enamel. *Journal of Dental Research* **71**, 20.

Titley KC, Torneck CD, Ruse ND, Krmec D (1993) Adhesion of a resin composite to bleached and unbleached human enamel. *Journal of Endodontics* **19**, 112–5.

Torneck CD, Titley KC, Smith DC, Adibfar A (1990) Adhesion of light cured composite resin to bleached and unbleached bovine dentine. *Endodontics and Dental Traumatology* **6**, 97–103.

Torneck CD, Titley KC, Smith DC, Adibfar A (1991) Effect of water leaching on the adhesion of composite resin to bleached and unbleached bovine enamel. *Journal of Endodontics* **17**, 156–60.

Youngson CC, Grey NJA, Jones JG (1990) In vitro marginal microleakage: examination of measurement used in assessment. *Journal of Dentistry* **18**, 142–6.