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The Microhardness of Bleached Dentine and its Bond Strength to a Dentine Bonding Agent

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Abstract - *The aim of this study was to measure the hardness of a bleached dentine surface and its bond strength to a dentine-bonding agent. Thirty teeth were randomly divided into a test and control group. The teeth were hemi-sectioned, the cut surfaces ground flat and the test surfaces bleached with a 10% aqueous solution of carbamide peroxide continuously for 4 days. Hardness was determined using a Vickers microhardness test. The bond between Coltene 'One Coat Bond' and bleached and unbleached dentine was evaluated by measuring shear bond strength using an Instron machine. The mean hardness of dentine before and after bleaching was 62.5 (10.2) and 53.6 (7.3) and this difference was statistically different ($p<0.001$). For the controls immersed in water the hardness was before 60.8 (standard deviation: 7.2) and after 59.6 (8.2) respectively. The mean shear bond strength for the unbleached was 5.5MPa (1.6) and for the bleached samples was 3.3MPa (1.8) and this difference was statistically significant ($p<0.002$). Under these study conditions the Vickers hardness and bond strength to dentine was reduced by bleaching.*

KEY WORDS: Vital bleaching, Carbamide peroxide, Dentine

INTRODUCTION

Improving the appearance of vital teeth with bleaching is well recognized. The consequences and sequelae of vital bleaching has been investigated and reported particularly with regard to the safety issues [1, 2]. The effects of 10% aqueous carbamide peroxide on enamel has been widely reported using scanning electron microscopy [3], hardness testing [4] and shear bond strengths [5]. The consensus being that any changes to the enamel surface is likely to be transient and provided bonding is delayed for at least 24 hours there should be no detriment to the survivability of the restoration [1]. However, there has been comparatively little research on the effects of 10% carbamide peroxide on dentine.

The techniques used to test hardness and bond strength vary between laboratories. The Vickers hardness test is a recognized method for measuring enamel or dentine but the machine was predominantly designed for materials other than teeth. Despite these difficulties the method has been widely reported [6, 7]. The possibility that bleaching might influence bond strength to enamel was reported by Haywood et al [3]. The authors suggested that a residual peroxide layer on enamel or oxygen from the hydrogen peroxide interfered with the polymerization of the resin and so reduced the bond strength. Since the time of that publication, dentine bonding agents have developed. Again, there is little information on the consequences of bonding to bleached dentine. There is some suggestion that a reduction in hardness occur [6] but generally little change probably occurs on the surface [1].

Dentine bonding agents link hydrophobic resins to the hydrophilic dentine surface. They form this bond partly from micromechanical retention and hybrid layer [8]. Manufacturers use different ways of forming this link. Some use volatile primers to treat the dentine surface whilst others incorporate water based materials to form the link between dentine and the composite. One coat bond has been comprehensively investigated and has shown comparable performance to other bonding systems [6-11].

The aim of this study was to investigate the effect of bleaching, with a 10% aqueous solution of carbamide peroxide, on the hardness and the tensile bond strength to dentine. The null hypothesis is that bleached dentine has a similar bond strength and microhardness as unbleached dentine.

METHOD AND MATERIALS

Thirty intact, caries-free teeth were collected, cleaned and sectioned longitudinally along the mesio-distal plane to form two halves of a tooth. Each half was labelled and the randomly assigned to the test or control groups. All sections were then embedded in self curing resin[a] and the cut surfaces ground with 240 and 600 grit silicone carbide papers until flat. The coronal surfaces of each specimen were inspected under a confocal microscope to ensure that the resin did not penetrate the dentine surface. The paired sections were randomly assigned to two further groups, 15 pairs for hardness testing and the remainder for bond strength assessment. The teeth were kept in distilled water at room temperature between investigations to prevent dehydration. The dentine surfaces of the test sections were immersed in a 10% bleaching gel[b] at pH 7, for 9 hours periods over 4 days. Sufficient bleaching gel was used during each immersion to completely cover the dentine sections. At the end of each session the sections

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were washed with distilled water and re-immersed. A new bleaching agent was used after each immersion period. The control samples were immersed in distilled water for the same period.

Hardness was assessed using a Vickers Hardness tester[c]. Three indentations were made in dentine with a 500g load for 10 seconds. The horizontal and vertical planes sections were measured and used to calculate the hardness of each indentation using calibration tables supplied by the manufacturer. A mean of the three indentations was used to calculate the hardness for that specimen.

The bond strengths between adhesives and the dentine were assessed with an Instron Universal testing machine[d]. Dentine samples were etched using 15% Phosphoric acid for 30 seconds, the tooth rinsed for 20 seconds and dried for a further 2 seconds using a 3 in-1-syringe as per the manufacturer's instructions. One coat bond[e] was then applied to the dentine for 20 seconds followed by a further 2 seconds of drying. The bonding agent was light cured for 30 seconds and Herculite XRV[f] built up by incremental curing to a 2mm long, 7mm diameter, standard shape using a silicone matrix. Each specimen was subject to one 100 N load with a cross head speed of 0.5mm/min at the composite/dentine interface until failure. Paired t-tests were used to compare the data for hardness and bond strengths.

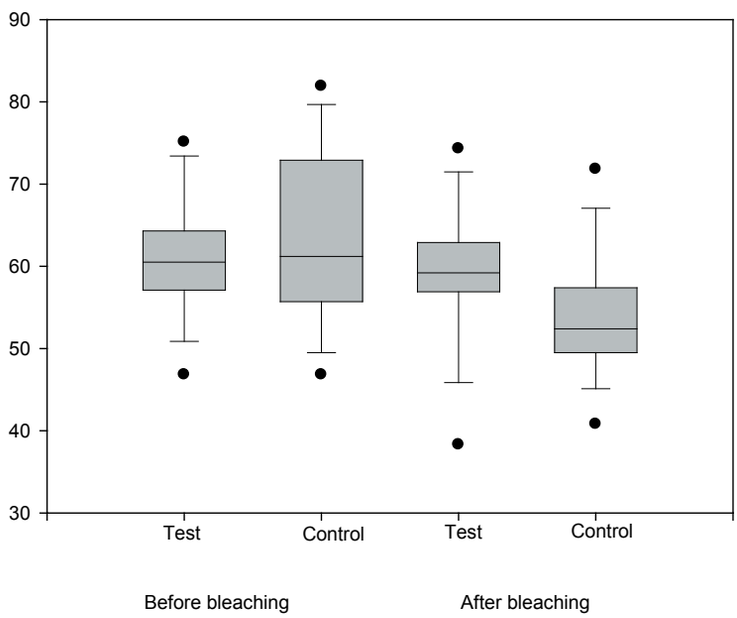

Figure 1. *Mean microhardness (standard deviation and 95% confidence intervals for the mean) of test dentine (Column 1) and control dentine (Column 2) and after immersion in carbamide peroxide (Column 3) or in water (Column 4).*

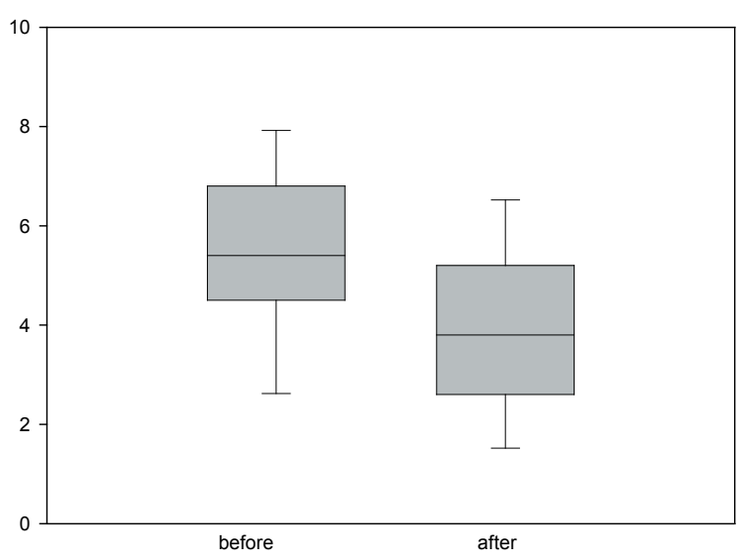

Figure 2. *Bond strength (MPa) of dentine before and after bleaching.*

RESULTS

The mean Vickers hardness of dentine (Figure 1) before and after bleaching were 62.5 (SD 10.2) and 53.6 (7.3) and for the unbleached controls before and after immersion in water was 60.8 (7.2) and 59.6 (8.2) respectively. There was no difference between the test and control before treatment ($p=0.4$), but hardness was significantly reduced after treatment ($p<0.001$). The mean shear bond strength (Figure 2) for the unbleached control was 5.5MPa (1.6) and for the bleached samples 3.3MPa (1.8). This difference, representing a 31% reduction in bond strength, was statistically significant ($p<0.002$).

DISCUSSION

The results of the hardness testing support previous work on enamel in that the hardness of dentine is affected by the bleaching process [1]. The reduction in hardness may result from the interaction between the hydrogen peroxide and the organic component of the dentine. This may alter the physical consistency of the dentine and provide a more elastic surface to the indentation from the Vickers machine**.** Demineralization of the tooth surface by a low pH solution would also affect the hardness value but the gel in this study was at neutral pH. A previous study has reported that hardness values return to the pre-bleached values after 14 days re-hydration [1,2].

Microhardness was determined using the Vickers equipment or Knoop have been reported to measure dentine hardness and followed previously published protocols [13]. The hardness testing revealed a reduction between the baseline and the bleached samples and follow a similar pattern to other investigations on dentine [14]. What effect saliva would have the hardness is not possible to predict as this was not done in this study. But some rehardening or repair of the surface might have been expected if saliva had been used [14].

Another aspect of the study design was that the effects of salivary remineralisation were not investigated. Clinically, saliva will continually bathe the dentine surface throughout any bleaching process and this may have influenced any results. Some information about the behavior of saliva on bleached dentine can be gained from previous investigations. Attin et al [15] showed a reduction in enamel hardness after bleaching returned to near normal levels following application of fluoride. Josey *et al* [16] and Murchison *et al* [4] used artificial saliva as a storage media in their studies to try and evaluate the potential for remineralisation following exposure to bleaching agents and to try and replicate the *in vivo* situation. Both studies found no significant difference between shear bond strengths of bleached and unbleached enamel.

The effect on the bond strength has clinical relevance. One study reported a similar proportional reduction in bond strength to dentine for 'Single Bond' and Z100 (3M products, UK) after bleaching [17]. Molla et al [18] reported higher bond strengths (19-39.9MPa) for unbleached dentine than we have reported in this study. This may reflect the different testing regimes and nature of the teeth used in the studies. The authors also used a wider diameter composite cylinder bonded to the dentine which accounts for the difference in bonding values. Sengun et al [19] suggested that sclerotic and demineralized dentine may be responsible for reduced bond strengths. The age and history of the teeth used in this study were unknown and there is a possibility that this factor may have influenced the results.

Every effort was made to standardise the cylinders of composite. The same silicone cylinder was used for each test. Some flexure of the long cylinder may have influenced the results but the length used in this study is less than that reported by others [19]. In so much as could be controlled the study design was kept as tightly controlled as possible to ensure that the same environment was present for each test. Perhaps repeated bond strengths on a number of samples may have been feasible but the previously bonded surface would need re sanding and therefore producing an unlike sample to the others. Finally, the samples were stored dry between the testing and the bonding but the time interval was very short, and almost instantaneous. It might have been interesting to investigate the influence of re-hydration either with saliva or water on the dentine following the bleaching to investigate this effect.

The results of this study, using paired samples from the same tooth, suggest that bleaching reduces the bond strength of a well known modern dentine bonding agent. The clinical implication of this is that extra caution should be shown when bonding to bleached dentine particularly if bonding occurs immediately after any bleaching is completed. Clinically, it remains a challenge to ensure the optimum bond between bleached dentine and a resin composite. More research is needed to evaluate the effect of time on this bond.

MANUFACTURERS DETAILS

- Duralay, Reliance Dental Mfg, Ill, USA
- Optident, West Yorkshire
- Vickers Hardness tester, York, UK
- Instron Universal testing machin, model 1193, Canton USA
- One coat bond· Coltene®, Switzerland
- Herculite XRV· Kerr/Hawes, Peterborough, UK

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REFERENCES

1. Dadoun MP, Bartlett DW. Safety issues when using carbamide peroxide to bleach vital teeth--a review of the literature. *Eur J Prosthodont Restor Dent* 2003; **11**:9-13.
2. Kelleher M, Roe FJC. The safety-in-use of 10% carbamide peroxide (Opalescence) for bleaching teeth under the supervision of a dentist. *Br Dent J* 1999; **187**:190-194.
3. Haywood VB, Leech T, Heymann HO, Crumpler D, Bruggers K. Nightguard bleaching:effects on enamel surface texture and diffusion. *Quintessence Int* 1990; **21**:801-804.
4. Murchison DF, Charlton DG, Moore BK. Carbamide peroxide bleaching: effects on enamel surface hardness and bonding. *Oper Dent* 1992; **17**:181-185.
5. Stokes AN, Hood JA, Dhariwal D, Patel K. Effect of peroxide bleaches on resin-enamel bonds. *Quintessence Int* 1992; **23**:769-771.

6. Lewinstein I, Hirschfield C, Stabholz A, Rotstein I. The effect of hydrogen peroxide and sodium perborate on the microhardness with enamel and dentine. *J Endodont* 1994; **20**:61-63.
7. Cimilli H, Pameijer CH. Effect of carbamide peroxide bleaching agents on the physical properties and chemical composition of enamel. *Am J Dent* 2001; **14**:63-66.
8. Watson TF, Bartlett DW. Adhesive systems: composites, dentine bonding agents and glass ionomers. *Br Dent J* 1994; **176**:227-231.
9. Li H, Burrow MF, Tyas MJ. Nanoleakage of cervical restorations of four dentin bonding systems. *J Adhes Dent* 2000; **2**:57-65.
10. Li H, Burrow MF, Tyas MJ. Nanoleakage patterns of four dentin bonding systems. *Dent Mater* 2000; **16**:48-56.
11. Brunton PA, Kalsi KS, Watts DC, Wilson NH. Resistance of two dentin-bonding agents and a dentin densensitizer to acid erosion in vitro. *Dent Mater* 2000; **16**:351-355.
12. de Freitas PM, Basting RT, Rodrigues JA, Serra MC. Effects of two 10% peroxide carbamide bleaching agents on dentin microhardness at different time intervals. *Quintessence Int* 2002; **33**:370-375.
13. Bartlett DW, Smith BG, Wilson RF. Comparison of the effect of fluoride and non-fluoride toothpaste on tooth wear in vitro and the influence of enamel fluoride concentration and hardness of enamel. *Br Dent J* 1994; **176**:346-348.
14. Attin T, Vollmer D, Wiegand A, Attin R, Betke H. Subsurface microhardness of enamel and dentin after different external bleaching procedures. *Am J Dent* 2005; **18**:8-12.
15) Attin T, Kielbassa AM, Schwanenberg M, Hellwig E. Effect of fluoride treatment on remineralization of bleached enamel. *J Oral Rehabil* 1997; **24**:282-286.
16. Josey AL, Meyers IA, Romaniuk K, Symons AL. The effect of a vital bleaching technique on enamel surface morphology and the bonding of composite resin to enamel. *J Oral Rehabil* 1996; **23**:244-250.
17. Spyrides GM, Perdigao J, Pagani C, Araujo MA, Spyrides SM. Effect of whitening agents on dentin bonding. *J Esthet Dent* 2000; **12**:264-270.
18. Molla K, Park HJ, Haller B. Bond strength of adhesive/composite combinations to dentin involving total- and self-etch adhesives. *J Adhes Dent* 2002; **4**:171-180.
19. Sengun A, Unlu N, Ozer F, Ozturk B. Bond strength of five current adhesives to caries-affected dentin. *J Oral Rehabil* 2002; **29**:777-781.