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Initial and fatigue bond strengths of chromatic and light-cured adhesives

June M.L. Lee, George Georgiou and Steven P. Jones
UCL Eastman Dental Institute, London, United Kingdom

Aim: To compare the initial and fatigue shear bond strengths of a chromatic adhesive with a light-cured adhesive in an ex vivo laboratory study.
Methods: Hydroxyapatite discs were used as the bonding substrate. They were produced by cold uni-axial compression at 20 tons, sintered at 1300 °C and embedded in epoxy resin before grinding and polishing. One hundred and fifty upper left central incisor brackets were bonded to the discs with Transbond PLUS Color Change (3M Unitek, Monrovia, CA, USA) while another 150 similar brackets were bonded with Transbond XT (3M Unitek, Monrovia, CA, USA). Seventy-five brackets from each group were subjected to cyclic loading (5000 cycles at 2 Hz) at 50 per cent of the mean bond strength in a Dartec Series HC10 Testing Machine. Initial (unfatigued) and fatigued bond strengths were determined by applying a shear force at the bracket-substrate interface using a custom-made metal jig in an Instron Universal Testing Machine. One-way ANOVA with Bonferroni post-hoc correction and two-way ANOVA were used to analyse the differences between the initial and fatigue mean shear bond strengths of the adhesives. The survival and bond reliability of both adhesives were evaluated with the Kaplan-Meier and Cox regression analyses.
Results: The initial mean shear bond strength for Transbond PLUS Color Change (16.72 MPa) was higher than Transbond XT (15.11 MPa), but this was not statistically significant ($p = 0.109$). The fatigue mean shear bond strength for Transbond XT (15.87 MPa) was similar to that of Transbond PLUS Color Change (15.33 MPa), and the difference was not statistically significant ($p > 0.999$). There were no significant differences when the effects of the material ($p = 0.264$) or fatiguing ($p = 0.512$) were considered separately, but in combination, the effect on bond strength was statistically significant ($p = 0.026$). The survival analysis showed that both adhesives demonstrated similar survival patterns in the unfatigued and fatigued states. Analysis of the material type and fatiguing showed no effect on the survival pattern for both adhesives ($p = 0.098$).
Conclusions: There were no statistically significant differences between the mean initial (unfatigued) and fatigue bond strengths of Transbond XT and Transbond PLUS Color Change under laboratory conditions. A survival analysis for both resins with and without fatigue loading exhibited similar behaviour with respect to their survival patterns. Although this may imply that under clinical conditions the two adhesives could behave similarly, the clinical extrapolation of these results should be interpreted with caution.
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June M. L. Lee: june_lee21@hotmail.com
George Georgiou: g.georgiou@eastman.ucl.ac.uk
Steven P. Jones: s.jones@eastman.ucl.ac.uk

Introduction

A clinically effective bonding adhesive should provide a secure bond between an orthodontic attachment and the tooth surface during treatment, but then allow removal of the attachment at the end of treatment without damaging the enamel. The search for the ideal bonding material in orthodontics has been the focus for much research and development, leading to the introduction of newer bonding materials to the market. In addition to any new properties, an adhesive must also retain the strengths found in current adhesives used by clinicians for it to be a viable substitute. The process of developing a new bonding agent relies on effective ex vivo laboratory studies which are commonly carried out prior to clinical trials, although comparisons between laboratory studies can be fraught with difficulties due to variations in their methodology and protocols. This has led to the emphasis on the importance of standardised test parameters and protocols.[1,2]

During fixed appliance therapy, patients may be at a significant risk of developing white spot lesions if oral hygiene is suboptimal.[3,4] Chromatic adhesives were developed to aid the removal of flash adhesive during bonding, since it has been suggested that the removal of flash may reduce plaque accumulation and the incidence of enamel demineralisation. These adhesives may either be photochromatic, where the adhesive turns clear on exposure to the curing light; or thermochromatic, where the adhesive turns clear above 32 °C and then reverts to its original colour below 32 °C to allow complete removal at debonding. Transbond PLUS Color Change adhesive (3M Unitek, Monrovia, CA, USA) is a light-activated photochromatic composite resin that changes from a pink-coloured resin to a tooth-coloured resin upon curing. The manufacturer claims that the colour changing property aids bracket positioning and flash clean-up around brackets, although this was not supported by a typodont study which found that the use of chromatic adhesive did not result in improved removal of excess adhesive and that significant amounts of adhesive flash remained after bonding.[5] There is limited published data relating to the bond strength of this material.[6,7]

Ex vivo laboratory bonding studies have traditionally used extracted human premolar teeth as the bonding substrate and the shear bond strengths of brackets bonded to human enamel has been reported to be in the range of 15–20 MPa.[8–10] The use of extracted human teeth has potential disadvantages which include the inconsistencies of crown contour and morphology, presence of surface defects and restorations, variations in fluoride mineralisation, coupled with an increasing difficulty in collecting suitable teeth for adequate sample sizes and problems related to the storage and sterilisation of human teeth. A previous study considered alternative substrates for the mineral phase of enamel, thereby introducing a biomimetic approach to laboratory bond strength testing. The use of cold-pressed commercially available pure hydroxyapatite powder has been recommended as an alternative substrate to enamel for comparative laboratory studies.[11,12]

Fatigue can be defined as 'degradation of materials subjected to a number of load changes, with a tendency to fracture under cyclic stress'. Fatigue failure is a phenomenon whereby stress values well below the ultimate tensile or shear stress of a material can produce premature fracture, because microscopic flaws grow slowly over many cycles of stress.[13] The structure eventually fails after being repeatedly subjected to loads that are so small that one application apparently causes no damage. Fatigue may be caused by thermal insults or by mechanical means. During fixed appliance therapy, bonded attachments may be subjected to repeated loading from masticatory forces or via the archwires. These forces vary in frequency, duration and magnitude, and when repeated over long periods of time may eventually result in structural or fatigue failure. It is important that when selecting a particular bonding material for clinical use, the fatigue resistance of the material is considered, as it will affect the durability of bonded attachments throughout the course of treatment.[14]

The conventional method for comparing two bonding adhesives has been a comparison of their initial bond strengths. However, fatigue testing is a more appropriate test to investigate the survival and durability of the bonding adhesive in the mouth, since the life expectancy of an adhesive is influenced by cyclic loading from occlusal forces. Fatigue failure could be a factor in predicting the bond strength and eventually the long-term survival of the bonding adhesive in a clinical environment.[15]

This study was designed to compare the initial (unfatigued) and fatigue bond strengths of Transbond PLUS Color Change with Transbond XT in a laboratory setting. The results of this study will form a foundation for future in vivo clinical trials.

Materials and methods

Circular hydroxyapatite discs were manufactured from commercially available hydroxyapatite powder Captal R (Plasma Biotal Ltd., Tideswell, UK) by cold-pressing at 20 tons in a hydraulic press. These were sintered at 1300 °C in a furnace, allowed to cool to room temperature overnight and then embedded in acrylic resin before being polished to a standard protocol. The manufacturing and polishing protocols have been described in detail.[11,12]

A small pilot study confirmed that a sample size of 75 per test group would provide 80 per cent power

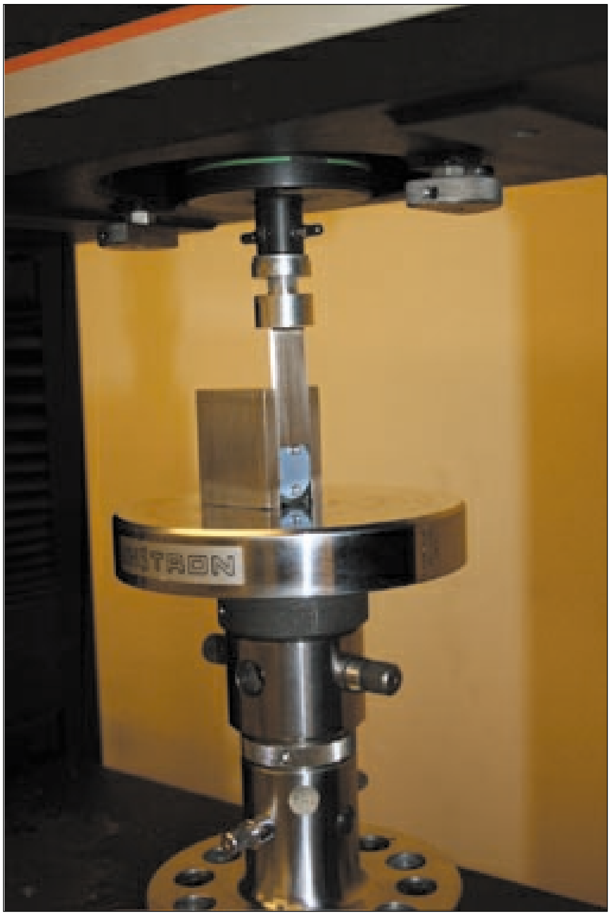

Figure 1. Jig mounted in Instron for bond strength testing, showing two opposing brackets in position.

Figure 2. Jig mounted in Dartec Series HC10 for cyclic fatigue loading, showing two opposing brackets in position.

(α = 0.05). Three hundred upper left central incisor, Victory Series stainless steel brackets (3M Unitek, Monrovia, CA, USA) were bonded to the hydroxyapatite discs with either Transbond XT (N = 150) or Transbond PLUS Color Change (N = 150) adhesives. The hydroxyapatite discs were etched with 35 per cent ortho-phosphoric acid gel prior to bonding, using a standardised bonding protocol.[11,12] Four brackets were bonded peripherally on each disc in order to allow the disc to be used for four separate shear tests. These were bonded in two stages using only opposing pairs of brackets at any one time to avoid dislodging the other brackets during testing.

The Instron Universal Testing Machine 4505 (Instron Limited, High Wycombe, UK) was used to carry out the shear bond strength testing of the bonded brackets. During testing each hydroxyapatite disc was held securely in place in the machine with a specially constructed metal jig (Figure 1). The load cell used for shear bond testing was 1 kN, at a cross-head speed of 1 mm per minute. Shear force was applied to the bracket-disc interface until the bracket failed. The maximum load at failure was recorded in kilonewtons (kN) and then converted to shear bond strength in megapascals (MPa) by dividing the maximum load at failure by the cross-sectional surface area of the bracket base.

Fatigue loading of bonded orthodontic brackets was carried out in the Dartec Series HC10 (Zwick/Roell, Herefordshire, UK). The cyclic mode of loading in this machine is designed to simulate repetitive occlusal forces to which the orthodontic brackets would be exposed intra-orally. The cyclic load applied was 50 per cent of the mean bond strength suggested by the pilot study. The loading cycle was 5000 cycles with the load applied in the form of a sine wave at a frequency of 2 Hertz (Figure 2). Once the fatigue cycle was completed, the bracket was then sheared to

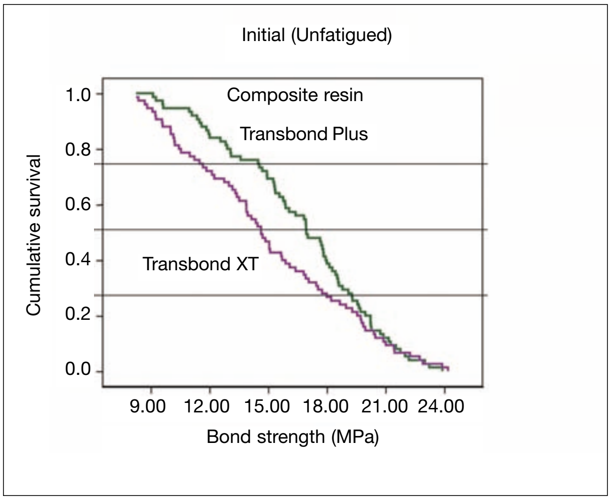

Figure 3. Kaplan-Meier plot for unfatigued brackets.

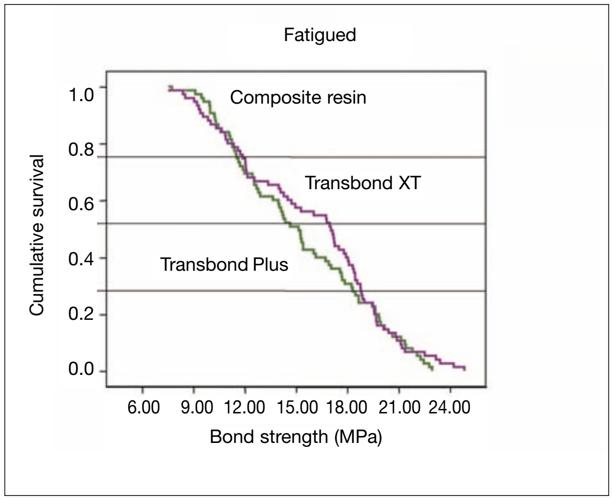

Figure 4. Kaplan-Meier plot for fatigued brackets.

failure in the Instron machine in order to determine the bond strength after fatigue cyclic loading.

As suggested from the sample size calculation, 75 brackets bonded with Transbond XT Light Cure Orthodontic Adhesive and 75 brackets bonded with Transbond PLUS Color Change Adhesive were sheared to failure without fatigue loading (initial bond strength test) and the same number of brackets for both adhesives were sheared to failure after fatigue loading.

A modified Kaplan-Meier survival curve was produced by plotting the cumulative survival probability against the bond strength to failure for the bonded brackets. This is a useful survival analysis tool to determine the bond reliability and survival probability at specific loads. A Cox regression analysis was used to investigate the simultaneous effect of a number of explanatory variables on survival.

Results

The mean shear bond strengths for all test groups are summarised in Table I. The data were found to be normally distributed and so parametric analyses were carried out. The mean differences in the shear bond strengths between the test groups were compared with a one-way ANOVA adjusted with the Bonferroni post-hoc correction for multiple comparisons. Transbond PLUS Color Change had a higher mean shear bond strength (16.72 MPa) than Transbond XT (15.11 MPa), but this was not statistically significant ($p = 0.109$). Transbond XT had a higher mean shear bond strength after fatiguing (15.87 MPa) than Transbond PLUS Color Change (15.33 MPa), but again this was not statistically significant ($p > 0.999$). Fatiguing produced no statistically significant differences in shear bond strength for either adhesive (Transbond PLUS Color Change: $p = 0.248$; Transbond XT: $p > 0.999$).

A two-way ANOVA was used to investigate the influence on bond strength of changing a single variable (the composite type or the introduction of fatigue), and whether there were any interactions when the composite type and fatigue were combined. There were no statistical significances when considering either the composite resin ($p = 0.264$) or the effect of fatiguing on the bonded brackets ($p = 0.512$). However, when composite type and fatigue were analysed in combination, the effect on bond strength was statistically significant ($p = 0.026$).

Figures 3 and 4 show modified Kaplan-Meier survival plots for unfatigued brackets and fatigued brackets respectively, for both composite resins. The Y-axis represents cumulative survival, such that a survival of 1.0 is 100 per cent survival, and 0.25 represents 25 per cent survival. Horizontal lines are drawn to highlight 25, 50 and 75 per cent survival. The profile plots for all test groups were comparable and this was confirmed by the Cox regression analysis that found no statistical significance between all the variables on the survival of the brackets. There were

Table I. Shear bond strength values (MPa) for test groups.

Group	N	Mean	95% CI	SD	Minimum	Maximum	SE
Transbond XT Initial	75	15.11	14.13, 16.10	4.29	8.26	24.19	0.49
Transbond PLUS Initial	75	16.72	15.87, 17.56	3.66	9.13	23.91	0.42
Transbond XT Fatigued	75	15.87	14.85, 16.89	4.42	7.54	24.85	0.51
Transbond PLUS Fatigued	75	15.33	14.38, 16.29	4.15	7.75	22.97	0.48

no statistically significant differences between the survival plots when considering the effects of the material (Transbond XT versus Transbond PLUS Color Change, $p = 0.184$), initial bond strength versus fatigue bond strength ($p = 0.290$) or the interaction between material and fatigue status ($p = 0.098$).

Discussion

The bonding technique used in this study was carried out in a dry field area following the manufacturer's instructions. A strict bonding protocol was adhered to for each bracket to ensure that every attempt was made to standardise the variables during the bonding process.[1,2] Polishing the enamel surfaces of the teeth with pumice is a common clinical practice before acid etching commences. It is also a common procedure in laboratory studies that have used human or animal teeth in bond strength testing. In this study, the hydroxyapatite discs were not pumiced because the disc surfaces had been ground and polished following a meticulous protocol to produce a uniformly polished surface. These surfaces were not covered by an organic pellicle which has been shown to cause poor bonding to enamel surfaces intra-orally.[16] Pumicing the hydroxyapatite surfaces would have offered no advantage and instead could become an unnecessary variable in this comparative bonding study.

One of the parameters in bond strength testing, which shows large inter-study variation, is the rate of force application as determined by the crosshead speed of the testing machine. Previous work has shown that varying the crosshead speed between 0.1 mm/min and 5.0 mm/min did not significantly influence either the debonding forces or the mode of failure of the bonded brackets.[17] In this study, a crosshead speed of 1.0 mm/min was used which fell within the previously proposed acceptable range and permitted an acceptable laboratory time per test. The load application chosen to debond the brackets was a shearing mode as this has been described to more closely resemble the occlusal forces exerted on bonded brackets intra-orally.[18–20] For the purpose of this study, a custom-made jig was used to hold the embedded hydroxyapatite discs firmly while the sliding blade directed the applied force vertically and as close as possible to the hydroxyapatite – adhesive interface.[11,12] The placement of the samples in the jig was carried out with great care to ensure that the direction of the applied force and the point of application of the force were consistent in all the samples. This is important because changes in the location of the applied force can cause significant differences in shear bond strength measurements and the failure pattern of the bond.[21]

The cyclic loading of the bonded brackets to simulate fatigue was carried out using a Dartec Series HC10, which is a hydraulic stress and strain testing machine. The fatigue load applied to the brackets was an estimation based on the initial bond strength from the pilot study. No standard force level or specific frequency of force application has been established to represent the forces to which bonded orthodontic brackets are subjected during a course of treatment. In this study, the fatigue load was set at 50 per cent of the mean bond strength and loaded for 5000 cycles, so that brackets were not debonded during fatiguing. This was deemed to be adequate to represent masticatory forces ranging from 40 to 120 N.[18] This technique of testing the shear bond strength after a fixed number of fatigue cycles mirrored that of a previous study.[22]

In order for meaningful comparisons to be made between different studies, the materials and methodology of the studies should be identical.[1] These should include parameters such as the type of bracket and resin used, as well as the bonding and testing protocols. In this current study, the methods employed in the fabrication of the hydroxyapatite discs, the bonding and debonding protocols were similar to two reported studies.[11,12]

Table II. Survival percentiles for all test groups.

Initial/Fatigued	Composite Resin	25 per cent		50 per cent		75 per cent	
		Estimate	SE	Estimate	SE	Estimate	SE
Initial	Transbond XT	18.67	0.966	14.64	0.534	11.50	0.852
	Transbond PLUS	19.57	0.629	16.96	0.654	14.50	0.960
	Overall	19.29	0.454	15.86	0.601	12.84	0.550
Fatigued	Transbond XT	19.01	0.462	17.04	0.953	11.90	0.523
	Transbond PLUS	18.67	0.832	15.17	0.554	11.55	0.344
	Overall	18.89	0.426	15.43	0.865	11.74	0.291
Overall	Overall	19.01	0.308	15.78	0.502	12.02	0.319

The mean initial (unfatigued) bond strength value for Transbond XT bonded to hydroxyapatite was found to be 15.11 MPa. This was lower than the value found in previous studies.[11,12] However, the mean shear bond strength of Transbond XT in the present study still lies within the accepted range of 15–20 MPa for shear bond strength of brackets bonded to enamel.[8–10] Although the methodology was identical, the differences between the bond strengths from this study and previous studies may reflect the fact that the brackets used were from different manufacturers.[11,12]

The mean initial bond strength for Transbond PLUS Color Change was found to be 16.72 MPa. There are limited studies in the literature on the bond strength of Transbond PLUS Color Change and to date there is no published study of this bonding adhesive to hydroxyapatite to enable reliable comparison with the findings of this study. In the studies that have investigated the bonding strength of Transbond PLUS Color Change, the substrate, the bonding conditions and protocols were diverse. Bonding in dry and wet conditions with various primers on bovine enamel was used in one study,[7] while human premolars bonded in a dry field have been reported in another.[6]

The manufacturer claims that the bond strength of Transbond PLUS Color Change is comparable to the conventional bonding adhesive Transbond XT. In this study, the difference in bond strengths between the two adhesives was not statistically significant ($p = 0.109$). This supported the findings of Vicente et al. who found no significant difference between the bond strengths of these two adhesives when bonding was performed in dry conditions with no contamination.[7] However, when Transbond PLUS Color Change was used in wet conditions with moisture tolerant or self-etching primers, the bond strengths were significantly higher than Transbond XT. It was suggested by the authors that this could be due to a lower content of hydrophobic (Bis-GMA) monomer in Transbond PLUS Color Change and the addition of polyethylene glycol dimethacrylate which renders it less sensitive to wet conditions.[7]

It has been postulated by researchers that the mean bond strength values of bonded brackets after fatigue loading would be lower compared to the unfatigued brackets. This was based on the assumption that fatigue loading may cause, or at least influence the process of mechanical failure of the substrate-adhesive-bracket interfaces. In this study there were no statistically significant differences in bond strength for either adhesive following fatiguing (Transbond PLUS Color Change: $p = 0.248$; Transbond XT: $p > 0.999$).

The two variables, composite resin and fatigue loading, were analysed using a two-way ANOVA to investigate whether these variables influenced the shear bond strength of the bonded brackets. When the composite resin type alone was tested, it did not show a significant effect on the bond strength ($p = 0.264$). This suggested that the differences in the material type, which may include factors such as composition, physical and chemical properties of the materials per se, did not influence the shear bond strength. This was also demonstrated when fatigue loading was analysed in isolation ($p = 0.512$) suggesting that fatigue alone did not significantly affect the shear bond strength. However, when ANOVA analysed the two variables in combination, the interaction between the material type and fatigue loading was found to be statistically significant ($p = 0.026$). This may imply that the interaction could influence the bond strength by acting synergistically.

Survival analysis, such as the Weibull or Kaplan-Meier analysis, has been proposed by previous authors for orthodontic bond strength studies to provide more information for the clinician on the survival and reliability of the bonded brackets.[1,23] These analyses focus on the bond strength values at the lower end of the range (tail end of the distribution) which are more critical in the assessment of the probability of failure of the bonded bracket. This helps clinicians to evaluate whether a particular bonding system might perform safely and successfully when used clinically. The shear bond strength data from this study was subjected to a modified Kaplan-Meier survival analysis. The estimated bond strengths at the 25 per cent, 50 per cent and 75 per cent survival percentiles for all test groups are shown in Table II. For brackets that were bonded with Transbond XT and not fatigued, at an estimated shear force of 11.50 MPa, 75 per cent of the brackets survived while 75 per cent of unfatigued Transbond PLUS Color Change survived a higher shear force of 14.50 MPa. When the brackets bonded to Transbond XT were subjected to fatigue loading, the mean shear force for 75 per cent survival was estimated to be 11.90 MPa and for Transbond PLUS Color Change, 11.55 MPa. This suggests that the probability of survival and behaviour of the fatigued brackets bonded with either type of adhesive are similar at the lower end of the shear force range.

Although laboratory findings should be extrapolated to the clinical environment with caution, a number of clinical implications can be considered from this comparative study. There was no significant difference between the mean initial shear bond strengths for Transbond XT and Transbond PLUS Color Change. This suggests that the changes in constituents associated with the colour change material have resulted in no deterioration in bond strength performance. Neither material demonstrated significant differences in mean shear bond strength as a result of the fatigue process used in this study. The cyclic forces used may be considered as representative of the nature of loads produced by intra-oral masticatory forces. Both materials resisted these cyclic forces adequately and did not demonstrate significantly lower bond strengths as a result of fatigue. This suggests that the materials should resist intra-oral fatigue equally, with neither adhesive demonstrating superior performance over the other. The equality in performance of the two adhesives was reflected in the similarity of the Kaplan-Meier survival curves and lack of significant difference shown by the Cox regression. In order to confirm the findings of this laboratory study, it would be beneficial to follow this up with an in vivo clinical trial.

Conclusions

There was no statistically significant difference between the mean initial (unfatigued) bond strengths of Transbond XT and Transbond PLUS Color Change ($p = 0.109$).

There was no statistically significant difference between the fatigue bond strengths of Transbond XT and Transbond PLUS Color Change ($p > 0.999$).

There were no statistically significant differences between the initial and fatigue bond strengths for either Transbond XT ($p > 0.999$) or Transbond PLUS Color Change ($p = 0.248$).

A survival analysis for both composite resins with and without fatigue loading exhibited similar behaviour with respect to their survival patterns ($p = 0.098$).

In a laboratory setting, the shear bond strength of Transbond PLUS Color Change, a new chromatic adhesive, was comparable to Transbond XT in both fatigue and unfatigued conditions. Although this may imply that under clinical conditions the two adhesives could behave similarly, the clinical extrapolation of these results should be interpreted with caution.

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Corresponding author

Dr S. P. Jones
Orthodontic Unit
UCL Eastman Dental Institute
256 Gray's Inn Road
London, WC1X 8LD
United Kingdom
Tel: (+44 0) 20 7915 1068
Fax: (+44 0) 20 7915 1238
Email: s.jones@eastman.ucl.ac.uk

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