Evaluation of Degree of Cure and Shear Bond Strength of a Color Changing Light Cure Adhesive

Hanumantha Rao Chidipothu, Shyamala Chandrasekhar

ABSTRACT

Objective: To determine the efficiency of Transbond plus color change adhesive (TPCCA), a newly introduced orthodontic adhesive material with reference to shear bond strength (SBS), debonding characters and degree of cure (DC) and to correlate SBS to the DC.

Materials and methods: Forty human premolars, divided into Group I (n = 20) Transbond XT and Group II (n = 20) TPCCA were bonded with metal brackets using the adhesives. Brackets were debonded in shear on an Instron universal testing machine with a crosshead speed of 1 mm per minute. The mode of bond failure was determined by Modified ARI index and the DC was determined by FTIR analysis.

Results: There was no statistically significant (p > 0.05) difference between SBS of TPCCA [SD-8.87 (2.11)] and conventional Transbond XT [SD-10.54 (3.12)] and therefore was clinically acceptable. The Weibull analysis suggested that TPCCA behaved consistently with respect to bond failure and reliability. Both adhesives exhibited cohesive type of bond failure. A statistically significant lower percentage of DC was noted for TPCCA [65.9 (2.4)] than Transbond XT [68.7 (3.2)] but it was within the range reported in the literature (55-75%). Pearson’s correlation was significantly positive between SBS and DC for both adhesives.

Conclusion: TPCCA has optimum SBS, favorable debonding character and DC at clinically acceptable levels.

Clinical implication: TPCCA can be a considered as an innovative orthodontic adhesive material for clinical use.

Keywords: Shear bond strength, Degree of cure, Color change adhesive.

INTRODUCTION

Direct bonding of orthodontic brackets to the enamel surface has enhanced clinical practice of orthodontics in terms of esthetics, comfort, better oral hygiene, decreased gingival irritation and reduced chairside time. The pioneering work of Buonocore1 in 1955 on acid etch technique and the development of Bowens resin BIS-GMA (bis-phenol A glycidyl dimethacrylate) by Bowen2 in 1962 for restorative purpose made valuable contributions to direct bonding technique and were instrumental in developing new procedures and materials.

Transbond XT, a light-cured BIS-GMA based composite resin is a popularly used orthodontic adhesive agent with optimal bond strength for clinical practice.

Transbond plus color change adhesive (TPCCA) with special features of color change, moisture tolerance and fluoride release was recently introduced by manufacturer of Transbond XT (3M Unitek).3 According to manufacturer, its bond strength was comparable to Transbond XT adhesive. Color changing property enhanced bracket positioning and flash clean up. The adhesive initially appears pink, and after light curing the color fades away. However, the color change does not indicate curing of the adhesive. The TPCCA with its many added advantages according to manufacture, also needs evaluation.

Orthodontic adhesives should have clinically acceptable shear and tensile bond strength to withstand masticatory forces and those generated by treatment mechanics, at the same time permitting bracket removal without damage to enamel surface and easy clean up.

To a greater extent mechanical property of the adhesive, such as flexural modulus of elasticity, tensile strength and compressive strength, depend on the degree of cure (DC) of the resin matrix. The color stability, solubility, degradation and biocompatibility are also strongly correlated to the DC.4

One of the major problems with light cure composite resins is their incomplete polymerization.5

There have been many investigations on the leaching of these residual monomers and the adverse reactions caused. Biocompatibility aspects of various orthodontic adhesive resins have been examined, and both in vitro and in vivo
reactions have been reported, including the potential for mutagenic effects and estrogenicity. The latter has recently gained special interest in the light of new evidence that some resins release Bisphenol A, a BIS-GMA monomer precursor, which exhibits estrogenicity. The cytotoxic responses were well correlated with substances eluted from the polymer restorative materials. Research on water sorption, solubility and degradation of resin composites has also suggested that leachable substances may cause toxic responses.6-10

Fourier transform infrared (FTIR) spectroscopy has been proven to be a powerful technique and has been used as a reliable method as it detects the C = C stretching vibrations directly before and after curing of composite resins.5

Hence, the present study was planned to determine the clinical usefulness of TPCCA based on the shear bond strength (SBS), debonding character and degree of cure (DC) in comparison with conventionally used Transbond XT.

MATERIALS AND METHODS
Methodology for Assessment of Shear Bond Strength
Forty upper premolar teeth extracted for orthodontic purpose were collected. Inclusion criteria for the tooth selection included anatomically and morphologically well-defined upper first premolar teeth with intact buccal enamel, extracted for orthodontic purpose.

Exclusion criteria for the sample consisted of teeth with carries heavy restorations, variations in crown with enamel structural defects, fractured crowns, fluorosed teeth.

The selected teeth were cleaned and stored in solution of 0.1%. Weight/Volume of thymol solution until bonding. The teeth were then mounted on self-cured acrylic blocks, up to cemento enamel junction with the buccal surface of crown perpendicular to base of the block. The teeth were divided into Group I (n = 20) Transbond XT and Group II (n = 20) TPCCA. Forty preadjusted edgewise upper premolar stainless steel brackets (Gemini series 80 gauge mesh, Unitek) were used. The buccal surface of the teeth was polished with pumice slurry using rubber cup. After polishing, the teeth were etched with 37% orthophosphoric acid for a period of 15 seconds. The acid was then washed away with a spray of water for 10 seconds. The tooth surface was then air dried using oil and it on a yellow color ceramic slab with 75% reflectance and measurement of the adhesive on the base of the bracket and placing the primer on a black paper from the base of the brackets and from the debonded tooth surface was scanned using an EPSON Scanner attached to a Macintosh computer. The scanned image was then viewed under a resolution of 1,200 dpi (dots/inch). Modified ARI scores was used to determine adhesive remaining on the enamel.

Bonding using TPCCA—Group II

The procedure for bonding TPCCA was same as that of Transbond XT with reference to application of primer, positioning of bracket and curing.

The bonded specimens were stored in distilled water for 24 hours at room temperature before evaluation of bond strength.

Evaluation of Shear Bond Strength
Debonding was carried out with an Instron universal testing machine (Instron Corp - load cell= 1 Kilo Newton) with a crosshead speed of 1 mm/min.

The following formula was used to evaluate the SBS in MPa,

\[
\text{Shear bond strength (MPa)} = \frac{\text{Force in Newton}}{\text{Base area of the bracket (sq. mm)}}
\]

Evaluation of the Residual Adhesive
The debonded tooth surface was scanned using an EPSON Scanner attached to a Macintosh computer. The scanned image was then viewed under a resolution of 1,200 dpi (dots/inch). Modified ARI scores was used to determine adhesive remaining on the enamel.

Methodology for Assessment of Degree of Cure using the FTIR Spectrometer
The DC of both groups was estimated by subjecting the cured and uncured resin samples to FTIR analysis.

To evaluate the DC in most of the earlier studies the adhesive under consideration is cured by placing a known quantity of the adhesive on the base of the bracket and placing it on a yellow color ceramic slab with 75% reflectance and subjected to FTIR analysis.

However in this study, a modified procedure was adapted in which the cured adhesive bonded on to the etched enamel surface was scraped with BP blade (No.15) after debonding from the base of the brackets and from the debonded tooth surface which was carefully collected in a piece of black paper measuring 6 × 6 cm, folded, and kept in individual cellophane covers. Later they were transported and were analyzed by FTIR spectroscopy.

Measurement of Degree of Conversion
The degree of conversion of each specimen was determined by comparison of the aliphatic carbon = carbon with that of the aromatic component for the cured and uncured resins. The DC of each specimen was estimated on a relative percentage basis with the 2-frequency method and the tangent baseline technique. The aliphatic carbon = carbon double bond group has a characteristic infrared absorption peak around 1,636 cm⁻¹.
to 1,638 cm\(^{-1}\). The aromatic carbon = carbon single bond peaks around 1,605 to 1,608 cm\(^{-1}\) due to the aromatic bonds of the benzene rings in the monomer molecules. Aliphatic (C = C) bond stretching vibrations at 1,638 cm\(^{-1}\) were chosen as the analytic frequency, whereas the aromatic (C..C) bond stretching vibrations at 1,605 cm\(^{-1}\), which are not affected by the polymerization reaction, were selected as a reference frequency. The percentage of DC was then determined according to the residual double bonds (RDB).\(^{12}\)

\[
\% \text{ DC} = 100 \left(1 - \frac{\text{Cured aliphatic (C = C)}}{\text{Cured aromatic (C..C)}} \right) / \left(\frac{\text{Uncured Aliphatic (C = C)}}{\text{Uncured Aromatic (C..C)}}\right) \times 100
\]

Where,

- Cured aliphatic (C = C) = Absorption peak at 1,638 cm\(^{-1}\) of the cured specimen.
- Cured aromatic (C..C) = Absorption peak at 1,608 cm\(^{-1}\) of the cured specimen.
- Uncured aliphatic (C = C) = Absorption peak at 1,638 cm\(^{-1}\) of the uncured specimen.
- Uncured aromatic (C..C) = Absorption peak at 1,608 cm\(^{-1}\) of the uncured specimen.

The results obtained from the SBS testing, the modified ARI scores and DC for the two different adhesives, their mean and standard deviation were calculated and then subjected to statistical evaluation.

**STATISTICAL ANALYSIS**

Student t-test was done to determine if a significant difference existed between the two groups with reference to mean SBS. Mann-Whitney U test was used to determine the level of significance of modified ARI scores and DC between the two adhesive groups.

Pearson’s correlation was done to determine the type of correlation between SBS and DC of both adhesive groups.

**RESULTS**

It is evident from Table 1 that the mean SBS for TPCCA is lower than that of conventional Transbond XT. However, this difference was not statistically significant (p = 0.058).

The Weibull modulus (\(\beta\)) characteristic strength (\(\alpha\)) and the SBS values for probability of failure at 10 and 90% are shown in Table 2. The graphical representation of the Weibull curve is given in Graph 1.

The SBS for 10% probability of failure is almost same for Groups I and II (7 and 6 MPa) and will behave in a similar fashion.

However, 90% of bracket failure will occur at higher SBS (15 MPa) for Group I (Transbond XT) compared to Group II (Transbond plus color change) which will fail at 12 MPa.

**MODIFIED ARI INDEX**

Evaluation of the modified ARI score (Table 3) for the two groups revealed that the difference in mean modified ARI scores between Groups I and II is not statistically significant (p = 0.13).

Table 4 shows the difference in the percentage distribution of the modified ARI scores, of score two and three between the groups were minor and was not statistically significant (p = 0.22). Both the groups showed similar type of bracket failure.

**DEGREE OF CURE**

The mean DC of Group I was [68.7 (3.2)] higher than that of Group II [65.9 (2.4)] However, the difference between the two groups were statistically significant with p = 0.003 (Table 5).

Correlation analysis was done to establish the relation between SBS and DC for both Groups I and II. A significant positive correlation was evident between SBS and DC for Group I with \(r = +0.83, p = 0.000\) (Table 6, Graph 2) and for Group II with \(r = +0.62, p = 0.003\) (Table 6, Graph 3).

**DISCUSSION**

Acid etch technique originally introduced in restorative dentistry for enhancing the adhesion of restorative materials,
The continuous developments in dental materials has lead to further improvement in adhesive bonding formulations resulting in wide range of products including self-etching, moisture insensitive, fluoride releasing, antimicrobial primer systems, flowable composites to mention a few in search of an ideal orthodontic bonding adhesive system. TPCCA is yet another composite adhesive system introduced by 3M Unitek as a moisture tolerant light cure bonding system with special features of color change and fluoride release. The mean SBS of the TPCCA is lower than that of Transbond XT but higher than that of clinically acceptable range suggested by Reynolds (5.9-7.8 MPa). The difference between the two adhesive groups, however, was not statistically significant. There is no study reported in the literature regarding the bond strength of this new material for comparison excepting that of Vicente. Vicente et al evaluated the adhesive with focus on SBS and moisture tolerance property of TPCCA. SBS were evaluated under dry, water and saliva contaminated conditions using TSEP and TMIP. The bond strength reported was comparatively lower with a value ranging between 6.93 and 7.89 MPa under varying test conditions. The SBS values in the present study of TPCCA are higher than that of Vicente et al. The results, however, are not directly comparable because of different types of primers used. In the present study, conventional primers were used and Vicente et al used moisture tolerance primer. Vicente et al suggested use of TPCCA along with TSEP/TMIP if there is a risk of contamination with water or saliva. Northrup et al have reported clinically acceptable SBS of a similar color changing adhesive ‘bluegloo’ (Ormco Corp) with self-ligating bracket in comparison with Transbond XT bonded to self-ligating bracket and conventional stainless steel bracket. Bluegloo also posses the property of color change with temperature retaining blue color in cooler temperature and changes to tooth color with activation by light.

It is suggested that all bond strength testing should include some form of survival analysis. This gives the clinician a better idea of how the material (or) bracket is likely to perform in opened up new pathways for bonding brackets to the etched enamel surface. Evolution in orthodontic adhesive system has taken place in terms of composition of adhesives, modes of curing, enamel conditioning methods, types of primers with improved properties of moisture tolerance, fluoride release, biocompatibility, ease of handling, and reduced chairside time.
the clinical situation (probability of failure). The use of survival analysis, such as Weibull analysis, is more appropriate and has been applied in orthodontic bond strength testing.

Probability of failure (pf) is calculated based on Weibull modulus and characteristic strength. Weibull modulus ($\beta$) indicates the scatter of results and is an indication of dependability of the material. A high value of ($\beta$) indicates a close grouping of the result and better dependability of the material.

It can be seen that TPCCA had slightly higher Weibull modulus value of 4.70 compared to that of Transbond XT with Weibull modulus value of 3.94. This higher Weibull modulus value, though marginal, indicates a more consistent behavior of the material for bond failure at a given debonding force.

Weibull curves were generated with percentage of probability of failure as a function of different bond strength values. It is evident from Graph 1 that at bond strength of 1 MPa, none of the brackets will fail in both the groups. The percentage of failure increases gradually from 6 to 13 MPa with 100% failure occurring at about 17 MPa.

The probability of failure is almost same at the tail end of the values 0 to 6 MPa and 16 to 20 MPa for both the adhesives. The pattern of failure is slightly different in the mid values, suggesting there is a marginal difference in bond failure rate for TPCCA compared to Transbond XT.

Thus, it can be inferred from the present study that TPCCA had comparable SBS at clinically acceptable levels to that of widely used and clinically efficient Transbond XT.

Results suggest that in vivo TPCCA will behave consistently with respect to bond failure and reliability.

Debonding character and failure pattern of the TPCCA and Transbond XT based on ARI score shows no statistical difference in percentage distribution of modified ARI score.

The ideal ARI score is a subject of debate. Lesser the amount of the adhesive left on the tooth surface (modified ARI of 4 and 5) less intervention is needed to remove residual adhesive and clean up procedures.

The DC for cured and uncured samples of TPCCA and Transbond XT were evaluation by FTIR spectroscopy.

The mean percentage of DC for TPCCA is [65.9(2.4)] and for that of Transbond XT is [68.7(3.2)].

In the light cure system the extent of polymerization or DC depend on several factors, composition of the resin, photoinitiator concentration, transmission of light through the material, exposure of time, light intensity of the curing unit and filler volume fraction which can induce reflection of light by both filler particles and tooth structure interposed between light source and composite.

In the present study all the factors related to the light source were the same excepting the adhesives (Transbond XT and TPCCA). There is a slight difference in the composition and percentage of BIS-GMA between the two adhesives. Transbond XT has 14% BIS-GMA and 9% BIS-EMA, whereas TPCCA has less than 2% BIS-GMA and 5 to 15% polyethylene glycol dimethacrylate. The lower DC for TPCCA may be due to difference in the composition and may not be an indication for incomplete polymerization resulting in residual monomer. This DC for TPCCA (64-67%) and Transbond XT (67-68%) is within the range reported in the literature for composite resins (55-75%). However residual monomer and its possibility of leaching and its effect on possible cytotoxicity on cell cultures needs further evaluation.

DC has been evaluated for composite resins in restorative dentistry. There are only few studies reported on DC of Transbond XT and other BIS-GMA based resins but not for TPCCA. Gioka et al 2005 evaluated the DC of Reliance BIS-GMA based light cure adhesive and reported 47% which is comparatively lower than the present study.

Shinya et al 2008 evaluated DC for Transbond XT immediately after 15 minutes of polymerization and has reported a value 37% less than that of present study.

Orthodontic bonding investigations have mostly focused on bond strength assessment. So far there is no study reported correlating between SBS and DC for any orthodontic adhesive including TPCCA.

It has been reported that DC modulates the physical and mechanical properties of dental composites including tensile and compressive strength. Correlation analysis in the present study between SBS and DC for both TPCCA and Transbond XT has confirmed this observation on DC and mechanical strength. A significant positive correlation was evident for TPCCA ($r = +0.62$ and $p = 0.003$) and for Transbond XT ($r = +0.83$ and $p = 0.000$).

TPCCA can be a material of choice if special features like moisture tolerance, fluoride release, antibacterial properties and biocompatibility are added apart from their existing characteristics.

CONCLUSION

TPCCA has optimum SBS at clinical acceptable levels, a favorable debonding character in comparison with widely used Transbond XT.

A statistically significant lower percentage of DC was noted for TPCCA compared to Transbond XT. However, this lower percentage of DC for TPCCA (64-67%) compared to Transbond XT (67-68%) is within the range reported in the literature for composite resins (55-75%).

A significant positive correlation was also established between SBS and DC for both TPCCA and Transbond XT.

Thus, TPCCA can be considered as an innovative orthodontic adhesive material for clinical use.

REFERENCES

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