

Fourier-transform infrared spectroscopy/attenuated total reflectance analysis for the degree of conversion and shear bond strength of Transbond XT adhesive system

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Objective: The objectives of this study were to evaluate the degree of conversion (DC) for Transbond XT curing light of intensity 1,600 mW/cm² by using variable curing durations and to determine the effect of the tested curing durations adopted in the current experiment on shear bond strength of Transbond XT resin cement.

Materials and methods: A total of 85 orthodontic ceramic brackets (Victory series; 3M Unitek) were utilized in the current experiment. The bonding system used in the current study was Transbond XT Primer followed by Transbond PLUS Color Change Adhesive (3M Unitek) that cured for 3, 6, and 9 seconds. The method was done by polymerization of the adhesive under a ceramic bracket for 40 ceramic brackets. The other 45 brackets were divided into three groups (n=15) according to the curing time duration (3, 6, and 9 seconds). The bonded specimens in each group were debonded using a shear load applied at the bracket bases by the blades of an Instron universal testing machine (ElectroPlus E1000; Instron) and directed in an occlusogingival direction with a crosshead speed of 0.5 mm/min utilizing 50 kg load cell.

Results: One-way ANOVA revealed that 6 and 9 seconds curing by the Ortholux light cure scored significantly higher values when compared to the 3 seconds curing.

Conclusion: Curing the Transbond XT for 6 and 9 seconds recorded a significant improvement of bond strength and DC.

Keywords: orthodontics, FTIR/ATR, degree of conversion, bond strength

Introduction

Light-emitting diode (LED) system has been the system of choice to cure the orthodontic resins, due to its brief duration of curing orthodontic resin cements.¹

LED technology is used for the polymerization of light-initiated dental materials.² LEDs use junctions of doped semiconductors to generate light. LEDs have a lifetime over 10,000 hours and undergo little change in output over this time.³

In orthodontic treatment, it is extremely important to achieve the optimum bond strength between the bracket and the tooth surface in the shortest possible time to avoid accidental debonding.^{3,4} The physical and mechanical properties of composite resin depend on optimal degree of cure, which is directly related to the intensity of light and radiation exposure time.⁴

During the early development of LED-curing units, manufacturers recommended an exposure time of 20–40 seconds/tooth to the light emitted by conventional light-curing

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devices with an intensity of ~400 mW/cm². During the last few years, many LED systems were introduced to the market that are capable of generating light with an intensity of approximately 800–1,000 mW/cm²; moreover, light exposure time associated with the use of these modern LED systems was reduced to 10–15 seconds.⁴ The tested LED system in the current study has an output energy of 1,600 mW/cm², and its curing time may be as short as 3 seconds.

Fourier-transform infrared (FTIR) spectroscopy is a widely used technique for investigating materials in the gaseous, liquid, or solid phase. It is based on the interaction between electromagnetic radiation and natural vibrations of the chemical bonds among atoms that compose the matter.⁵ FTIR can detect the stretching vibrations of carbon–carbon double bonds involved in polymerization.⁶ The method typically utilizes the height ratio of the peaks corresponding to aliphatic (1,637 cm⁻¹) and aromatic (1,715 cm⁻¹) double bonds to determine the degree of conversion (DC). FTIR/attenuated total reflectance (ATR) is more versatile than transmission FTIR because it eliminates the need of KBr (NaCl) plates, sectioning of thin wafers, or cure of the material in a thin film form, which simplifies sample preparation and more importantly allows the resin to be cured under conditions closer to clinical cases. FTIR/ATR also has an edge on evaluating the DC over the hardness test as it was reported previously.^{6,7}

The objectives of this study were to evaluate the DC for Transbond XT curing light of intensity 1,600 mW/cm² by using variable curing durations and to determine the effect of the tested curing durations adopted in the current experiment on shear bond strength of Transbond XT resin cement.

Materials and methods

The materials used in this study are all presented in Table 1 along with their composition.

Specimens' preparation

A total of 85 orthodontic ceramic brackets (Victory series; 3M Unitek, Monrovia, CA, USA) were used in the current experiment. The bonding system utilized in the current experiment was Transbond XT Primer followed by Transbond PLUS Color Change Adhesive (3M Unitek) that cured for 3, 6, and 9 seconds. LED (Ortholux; 3M Unitek) with an output of 1,600 mW/cm² was utilized in the current experiment. The light curing unit was calibrated using Demetron™ LED radiometer for choosing the proper energy output to have stable and accurate results while curing the resins.

FTIR/ATR analysis

Transbond XT was applied to the base of 40 ceramic brackets, and then, it was firmly pressed on the crystal of FTIR/ATR (Thermo Scientific Nicolet iS5 FTIR Spectrometer; Thermo Fisher Scientific, Waltham, MA, USA) using a load of 10 g to standardize the thickness of Transbond XT.⁷ The excess amount of resin was removed before polymerization. The specimens were light cured by the LED unit for 3, 6, and 9 seconds (n=15). The LED unit was slightly touching the bracket and was perpendicular to the bracket while curing.

FTIR spectroscopy was performed using the ATR accessory. All measurements were obtained under the following conditions: resolution of 4 cm⁻¹ and four internal scans per reading.^{8,9} For each cured resin matrix, the same non-cured matrix served as the control (Figure 1).^{10,11}

Table 1 Materials used in this study

Materials	Ingredients	Percentage by weight
Transbond XT primer	Bisphenol A diglycidyl ether dimethacrylate	45%–55%
	Triethylene glycol dimethacrylate	45%–55%
	Triphenylantimony	<1%
	4-(Dimethylamino)benzeneethanol	<0.5%
	dl-Camphorquinone	<0.3%
	Hydroquinone	<0.03%
Transbond XT adhesive	2-Hydroxy-1,2,3-propanetricarboxylic acid reaction products with 2-isocyanatoethyl methacrylate	5%–15%
	Silane-treated glass	35%–45%
	Silane-treated quartz	35%–45%
	Polyethylene glycol dimethacrylate	5%–15%
	Silane-treated silica	<2%
	Bisphenol A diglycidyl ether dimethacrylate (bisphenol A-glycidyl methacrylate [<i>bis</i> -GMA])	<2%
3M Unitek etching gel	Water	55%–65%
	Phosphoric acid	30%–40%
	Amorphous silica	5%–10%

Between each set of monomer/polymer spectra, the crystal plate of the ATR accessory was cleaned with an absorbent paper and ethyl alcohol and then dried with an air blower, so that there would be no residues that may affect the new set of monomer/polymer spectrum measurements.¹²

The spectra of the monomers and their respective polymers were compared to determine the conversion rate of the double bonds into simple carbon bonds. The peaks were measured at the frequencies of $1,715/\text{cm}^{-1}$ (corresponding to the aromatic ring bonds) and $1,637/\text{cm}^{-1}$ (corresponding

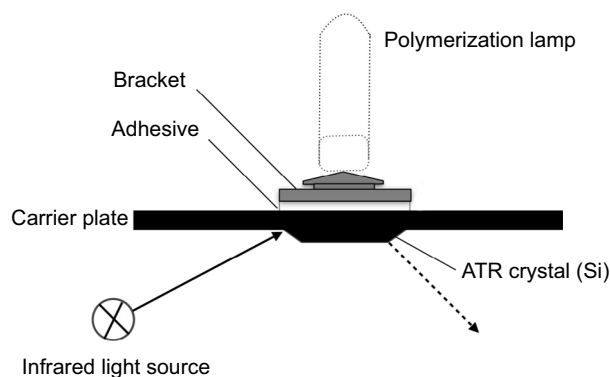


Figure 1 FTIR spectroscopy was performed using the ATR accessory and a plate of zinc selenite crystal slightly touching at 90° .

Note: All measurements were obtained under the following conditions: resolution of 4 cm^{-1} and four internal scans per reading.

Abbreviations: FTIR, Fourier-transform infrared; ATR, attenuated total reflectance.

to the bonds between carbons of the methacrylate groups; Figure 2). The following formula was used to calculate the conversion rate of the double carbon bonds into simple bonds:

$$\% \text{Conversion} = 100 \times 1 - \frac{\text{polymer (C=C)} \times \text{monomer (C-C)}}{\text{monomer (C=C)} \times \text{polymer (C-C)}}$$

Debonding of brackets

Based on 80% power of test, 45 sound extracted premolars, with no obvious enamel crack, were collected according to the guidelines approved by university and in accordance with the principles of the Declaration of Helsinki. Teeth were stored in distilled water, cleaned, polished with non-fluoridated pumice and rubber prophylactic cups, rinsed with water spray, and dried with oil-free compressed air. The remaining 45 ceramic brackets were bonded to 45 freshly extracted non-carious premolars teeth that were used.

Unitek™ Etching Gel (REF 712-0; 3M Unitek) was applied exclusively to the bracket bonding area for 15 seconds followed by rinsing and drying for 15 seconds. The 45 orthodontic ceramic brackets were bonded to the labial surfaces of the aforementioned prepared labial surfaces of the teeth by Transbond XT Primer followed by Transbond PLUS Color Change Adhesive. The specimens were light cured by the LED unit for 3, 6, and 9 seconds ($n=15$). The LED unit was slightly touching the bracket and was perpendicular to the bracket while curing.

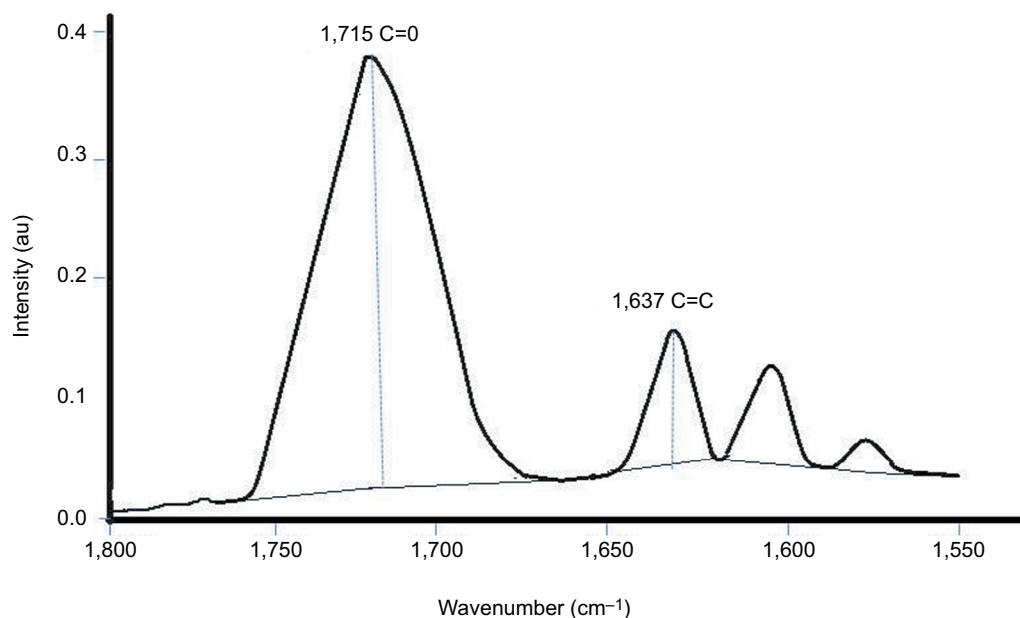


Figure 2 The spectra of the monomers and their respective polymers were compared to determine the conversion rate of the double bonds into simple carbon bonds.

Note: The peaks were measured at the frequencies of $1,715/\text{cm}$ (corresponding to the aromatic ring bonds) and $1,637/\text{cm}$ (corresponding to the bonds between carbons of the methacrylate groups).

Abbreviation: au, atomic unit.

The bonded specimens in each group were debonded using a shear load applied at the bracket bases by the blades of an Instron universal testing machine (ElectroPlus E1000; Instron, Canton, MA, USA; Figure 3) and directed in a gingival–occlusal direction with a crosshead speed of 0.5 mm/min utilizing 50 kg load cell.¹³

Statistical analysis

One-way ANOVA was used to detect the bond strength and the DC for the Transbond XT that was cured under orthodontic ceramic brackets for 3, 6, and 9 seconds.

Results

One-way ANOVA revealed that the curing duration in this study significantly affected the DC of the Transbond XT resin

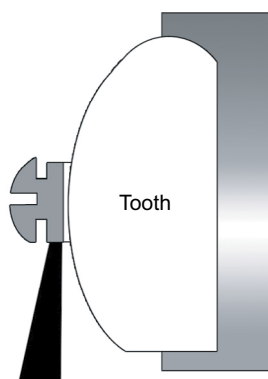


Figure 3 The bonded specimens in each group were debonded using a shear load applied at the bracket bases by the blades of an Instron Universal Testing Machine (ElectroPlus E1000; Instron, Canton, MA, USA) and directed in an occlusogingival direction with a crosshead speed of 0.5 mm/min utilizing 50 kg load cell.

cement and the bond strength of the tested ceramic brackets to human teeth ($P<0.05$).

Debonding of brackets

The 6 and 9 seconds curing by the Ortholux light scored significantly higher values when compared to the 3 seconds curing (recommended by the manufacturer), whereas the 9 seconds curing duration did not show a significant difference as shown in the 6 seconds curing ($P<0.05$; Figure 4).

DC

There was a significant increase in the DC when the Transbond XT was cured by the Ortholux curing unit for 6 seconds when compared to that for 3 seconds ($P>0.05$). Curing using the Ortholux curing unit showed no significant effect on the DC if the period of curing was increased to 9 seconds by the Ortholux curing unit. The 6 and 9 seconds curing by the Ortholux light scored significantly higher values when compared to the 3 seconds curing ($P<0.05$).

Discussion

Manufacturer's instructions to cure the Transbond XT under ceramic brackets is 3 seconds; however, our results showed that the curing duration of the Transbond XT under ceramic bracket using the Ortholux curing unit had a significant effect on the shear bond strength and the DC of the tested bonding system.

The composite resin used in this study was Transbond XT, which is one of the most popular resin cements used to

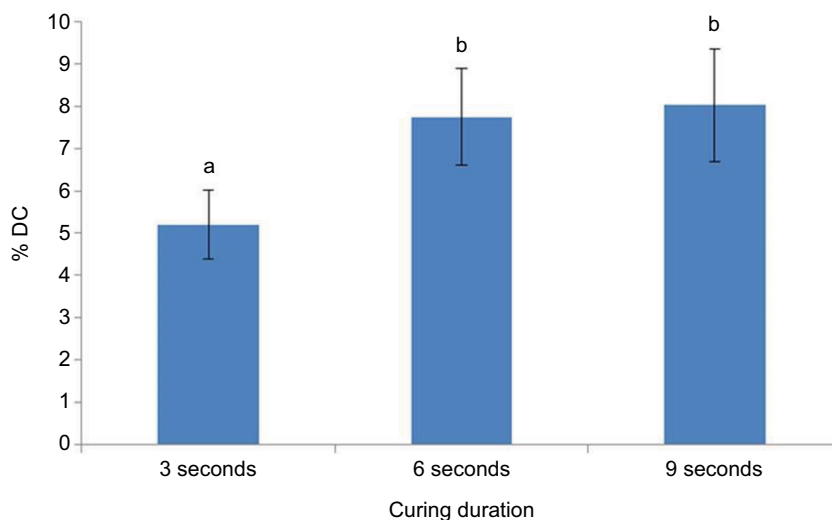


Figure 4 The 6 and 9 seconds curing by the Ortholux light scored significantly higher values when compared to the 3 seconds curing (recommended by the manufacturer), whereas the 9 seconds curing did not show a significant difference (Figure 2) when compared to the 6 seconds curing ($P<0.05$).

Abbreviation: DC, degree of conversion.

cement orthodontic brackets worldwide. The bond strength of Transbond XT to human teeth and its DC were examined because they are extremely important parameters that determine the physical and mechanical properties of various orthodontic bonding systems.¹⁴

The main aim of decreasing the recommended period for curing the orthodontic bonding systems under each orthodontic bracket is to decrease the chair time needed to bond a full set of orthodontic brackets. Previous researches have showed that curing the bonding system under each orthodontic brackets for 10 seconds/bracket by an Ortholux is equivalent in terms of clinical performance to curing the same bonding agent by halogen light curing systems for 40 seconds/bracket.¹⁵

Moreover, manufacturer of Ortholux recommended 3 seconds curing duration/bracket in case of using ceramic brackets depending on the permeability of the ceramic brackets to light. Our results showed that increasing the curing duration to 6 seconds/bracket will significantly improve the bond strength of the ceramic brackets to human enamel, which agrees with previous results that demonstrated an increase in bond strength of orthodontic metallic brackets to bovine enamel upon increasing the curing duration from 3 seconds to 6 seconds.

It was extremely difficult to compare the exact values of the bond strength obtained in the current study with the results obtained in previous studies due to the wide range of values recorded, ie, previous studies recorded values of 26.9 ± 8.1 MPa, while other studies recorded values of 8.4 MPa.^{16,17} A meta-analysis study suggested that the aforementioned wide range of bond strength values recorded was attributed to ~27 different parameters that may vary from one experiment to another according to the method adopted in each experiment, ie, direction of debonding force, load cell, crosshead speed, etc.¹⁸

The increase in bond strength values obtained in the current study may be explained by the recorded FTIR/ATR significant increase in the DC results upon increasing the curing duration of Transbond XT to 6 seconds because the DC is an important parameter in determining the final physical, mechanical, and biological properties of photo-activated composite resins. DC for resin cements is defined as the number of ethylene double carbon bonds that are converted into single bonds of the composite resin obtaining optimal chemical–physical properties, thereby increasing the clinical outcome. Hence, it plays a significant role in determining the ultimate success of the restoration.¹⁹ The DC is determined by the proportion of the remaining concentration of

the aliphatic C=C double bonds in a cured sample relative to the total number of C=C bonds in the uncured material.²⁰ Incomplete conversion may result in unreacted monomers, which might dissolve in the oral cavity, leading to the leach of various chemical compounds from the cured resin.²¹ In addition, reactive sites (double bonds) are susceptible to hydrolyzation or oxidation and, thereby, leads to a degradation of the material. Moreover, uncured functional groups can act as plasticizers, reducing the mechanical properties of the cured resin.

Although the current study reported only laboratory results, however, it may be suggested that there may be less incidence of debonding of early ceramic brackets upon curing Transbond XT under each ceramic bracket by Ortholux for 6 seconds, especially in cases of severe teeth crowding that place the orthodontic brackets and wires under high stresses directly after the bonding procedures.

Conclusion

Curing the Transbond XT for 6 or 9 seconds recorded a significantly improved bond strength and DC of Transbond XT bonding ceramic brackets to human enamel.

Ethics statement

a) Ethics Committee: The remaining 40 ceramic brackets were bonded to 40 freshly extracted non-carious premolar teeth that were used according to the guidelines approved by King Abdulaziz University and in accordance with the principles of the Declaration of Helsinki. The patients whose teeth had been extracted and used in this research provided written informed consent. The same consent for all procedures was obtained from all authors. All experimental procedures were done according to the guidelines of the ethical committee for reviewing experimental procedures at the Faculty of Dentistry, King Abdulaziz University.

b) All premolar teeth were sound teeth that were indicated for extraction as a part of an orthodontic treatment plan. All teeth were extracted in the Oral Surgery Department, Faculty of Dentistry.

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Disclosure

The authors report no conflicts of interest in this work.

References

1. Souza-Junior EJ, Prieto LT, Soares GP, Dias CT, Aguiar FH, Paulillo LA. The effect of curing light and chemical catalyst on the degree of conversion of two dual cured resin luting cements. *Lasers Med Sci.* 2012;27(1):145–151.
2. Aleksiejunaite M, Sidlauskas A, Vasiliauskas A. Effect of rebonding on the bond strength of orthodontic tubes: a comparison of light cure adhesive and resin-modified glass ionomer cement in vitro. *Int J Dent.* 2017;2017:6.
3. Cerveira GP, Berthold TB, Souto AA, Spohr AM, Marchioro EM. Degree of conversion and hardness of an orthodontic resin cured with a light-emitting diode and a quartz-tungsten-halogen light. *Eur J Orthod.* 2010;32(1):83–86.
4. Claudino D, Kuga MC, Belizário L, Pereira JR. Enamel evaluation by scanning electron microscopy after debonding brackets and removal of adhesive remnants. *J Clin Exp Dent.* 2018;10(3):e248–e251.
5. Garoushi S, Vallittu P, Shinya A, Lassila L. Influence of increment thickness on light transmission, degree of conversion and micro hardness of bulk fill composites. *Odontology.* 2016;104(3):291–297.
6. Ghavami-Lahiji M, Firouzmanesh M, Bagheri H, Jafarzadeh Kashi TS, Razazpour F, Behroozibakhsh M. The effect of thermocycling on the degree of conversion and mechanical properties of a microhybrid dental resin composite. *Restor Dent Endod.* 2018;43(2):e26.
7. Shinya M, Shinya A, Lassila LV, Varrela J, Vallittu PK. Enhanced degree of monomer conversion of orthodontic adhesives using a glass-fiber layer under the bracket. *Angle Orthod.* 2009;79(3):546–550.
8. Oliveira KM, Lancellotti AC, Ccahuana-Vásquez RA, Consani S. Shrinkage stress and degree of conversion of a dental composite submitted to different photoactivation protocols. *Acta Odontol Latinoam.* 2012;25(1):115–122.
9. Kambs B, Kettler J, Bock M, et al. Low-noise quantum frequency down-conversion of indistinguishable photons. *Opt Express.* 2016;24(19):22250–22260.
10. Cerutti F, Acquaviva PA, Gagliani M, et al. Degree of conversion of dual-cure resins light-cured through glass-fiber posts. *Am J Dent.* 2011;24(1):8–12.
11. Jafarzadeh TS, Erfan M, Behroozibakhsh M, et al. Evaluation of polymerization efficacy in composite resins via FT-IR spectroscopy and vickers microhardness test. *J Dent Res Dent Clin Dent Prospects.* 2015;9(4):226–232.
12. Alshali RZ, Silikas N, Satterthwaite JD. Degree of conversion of bulk-fill compared to conventional resin-composites at two time intervals. *Dent Mater.* 2013;29(9):e213–e217.
13. Linjawi AI, Abbassy MA. Comparison of shear bond strength to clinically simulated debonding of orthodontic brackets: An in vitro study. *J Orthod Sci.* 2016;5(1):25–29.
14. Yoshida S, Namura Y, Matsuda M, Saito A, Shimizu N. Influence of light dose on bond strength of orthodontic light-cured adhesives. *Eur J Orthod.* 2012;34(4):493–497.
15. Krishnaswamy NR, Sunitha C. Light-emitting diode vs halogen light curing of orthodontic brackets: a 15-month clinical study of bond failures. *Am J Orthod Dentofacial Orthop.* 2007;132(4):518–523.
16. Scougall Vilchis RJ, Yamamoto S, Kitai N, Yamamoto K. Shear bond strength of orthodontic brackets bonded with different self-etching adhesives. *Am J Orthod Dentofacial Orthop.* 2009;136(3):425–430.
17. Pamukcu H, Ozsoy OP, Dagalp R. *In vitro* and *in vivo* comparison of orthodontic indirect bonding resins: A prospective study. *Niger J Clin Pract.* 2018;21(5):614–623.
18. Finnema KJ, Ozcan M, Post WJ, Ren Y, Dijkstra PU. In-vitro orthodontic bond strength testing: a systematic review and meta-analysis. *Am J Orthod Dentofacial Orthop.* 2010;137(5):615–622.e3.
19. Souza RO, Ozcan M, Mesquita AM, et al. Effect of different polymerization devices on the degree of conversion and the physical properties of an indirect resin composite. *Acta Odontol Latinoam.* 2010;23(2):129–135.
20. Tonetto MR, Pinto SC, Rastelli AN, et al. Degree of conversion of polymer-matrix composite assessed by FTIR analysis. *J Contemp Dent Pract.* 2013;14(1):76–79.
21. Collares FM, Portella FF, Leitune VC, Samuel SM. Discrepancies in degree of conversion measurements by FTIR. *Braz Oral Res.* 2014;28:9–15.

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