©Operative Dentistry, 2007, **32-2**, 144-148

Influence of Curing Rate of Resin Composite on the Bond Strength to Dentin

AR Benetti • E Asmussen • A Peutzfeldt

Clinical Relevance

When deciding on a specific curing protocol, dental professionals should be aware of the advantages and limitations of each curing mode.

SUMMARY

This study determined whether the strength with which resin composite bonds to dentin is influenced by variations in the curing rate of resin composites. Resin composites were bonded to the dentin of extracted human molars. Adhesive (AdheSE, Ivoclar Vivadent) was applied and cured (10 seconds @ 1000 mW/cm²) for all groups. A split Teflon mold was clamped to the treated dentin surface and filled with resin composite. The rate of cure was varied, using one of four LED-curing units of different power densities. The rate of cure was also varied using the continuous or pulse-delay mode. In continuous curing mode, in order to give an energy density totaling 16 J/cm², the power densities (1000, 720, 550, 200 mW/cm²) emitted by the various curing units were compensated for by the light curing period (16, 22, 29 or 80 seconds). In the pulse-delay curing mode, two seconds of light curing at one of the four power densities was followed by a one-minute interval, after which light cure was completed (14, 29, 27 or 78 seconds), likewise, giving a total energy density of 16 J/cm². The specimens produced for each of the eight curing protocols and two resin composites (Tetric EvoCeram, Ivoclar Vivadent; Filtek Supreme XT, 3M ESPE) were stored in water at 37°C for seven days. The specimens were then either immediately subjected to shear bond strength testing or subjected to artificial aging (6,000 cycles between 5°C and 55°C baths) prior to testing. Failure modes were also assessed. The shear bond strengths were submitted to factorial analysis of variance, and the failure modes were submitted to a Chi-square test (α=0.05). All but power densi-

Ana Raquel Benetti, DDS, MS, PhD student, Department of Operative Dentistry, Endodontics and Dental Materials, Bauru School of Dentistry, University of São Paulo, Baru SP, Brazil

Erik Asmussen, MS, Dr Odont, professor, Department of Dental Materials, School of Dentistry, University of Copenhagen, Copenhagen N, Denmark

*Anne Peutzfeldt, DDS, PhD, Dr Odont, associate professor, Department of Dental Materials, University of Copenhagen, Copenhagen N, Denmark

*Reprint request: Nørre Allé 20, 2200 Copenhagen N, Denmark; e-mail: apz@odont.ku.dk

DOI: 10.2341/06-39

ty (curing mode, resin composite material and mode of aging) significantly affected shear bond strength. The curing mode and resin composite material also influenced the failure mode. At the selected constant energy density, pulse-delay curing reduced bonding of the resin composite to dentin.

INTRODUCTION

The degree of cure of a given polymeric material is directly related to the energy density received by the material: the higher the energy density, the higher the degree of cure, strength and stiffness of a resin composite.[1-5] However, a given energy density level may be obtained by different combinations of curing time and power density. There is evidence that increasing the power density, while maintaining constant energy density, decreases the degree of cure.[5] Furthermore, power density and cure temperature dramatically influence the kinetics of polymerization.[6]

The initial curing rate of a light-curing resin composite is influenced by the power density of the curing unit: a reduction in power density implies a reduction in the curing rate.[7] For a given energy density level, the curing reaction may also be slowed down by using so-called soft-start curing modes, which include step-curing and pulse-delay curing. Among these, in particular, the pulse-delay curing mode has been found to be effective in reducing the rate of cure as compared to continuous cure.[8]

The curing rate is related to stress formation in resin composites.[9] The remaining stresses in the tooth-restoration complex result in a higher risk of failure during function.[10] It was with the aim of minimizing the shrinkage stresses produced during curing of resin composites that soft-start curing modes were introduced. Slowing down the curing reaction by varying the curing mode has been found to minimize damage at the tooth-restoration interface.[11-13] Studies show that pulse-delay curing minimizes shrinkage stresses at the cavosurface interface,[13-14] diminishes post-gel polymerization shrinkage[15] and reduces contraction force during the initial phase of polymerization.[16] Since reducing the curing rate results in smaller remaining stresses in resin composites[9], one might speculate that reducing the curing rate may result in stronger bonds.

Only a few studies have investigated the effect of curing modes on the shear bond strength of resin composite to dentin. Price and others[17] found that varying curing modes by changing them from continuous to step-cure reduced the bonding of resin composite to dentin. Caldwell, Kulkarni and Titley,[18] however, did not observe significant differences between step-cure and continuous cure regarding bond strength. It should be noted that the energy density was not kept constant in either study.

While in service, the adhesive strength of restorations can be also challenged by thermal and fatigue stresses. Thermocycling is a widely accepted artificial mode of aging used to simulate alternating *in vivo* temperatures.[19] Although thermocycling does not always influence the mechanical properties of resin composite materials, it may result in the decreased bonding of resin composite to enamel[20] or dentin, particularly when increasing the number of cycles.[21] Since the curing rate and curing mode may affect the polymer structure,[22-24] thermocycling may have an influence on shear bond strength.

Therefore, the aim of this study was to determine whether the strength with which resin composite bonds to dentin is influenced by variations in the curing rate of the resin composite. The curing rate was varied: 1) by use of curing units operating at different power densities and 2) by the use of different curing modes. The null hypothesis for this experiment states that, at a given level of energy density, the curing rate does not influence the strength with which resin composite bonds to dentin.

METHODS AND MATERIALS

Shear Bond Strength

Human molars kept in 0.5% chloramine aqueous solution were embedded in epoxy resin. After setting, the mesial or distal surfaces were ground using sequential silicon carbide paper up to #1000 grit to expose a flat dentin surface. The dentin was treated with a self-etch adhesive (AdheSE, Ivoclar Vivadent, Schaan, Liechtenstein) following the manufacturer's instructions. The primer was applied for 30 seconds, the solvent evaporated with a strong stream of air and the adhesive was subsequently applied and light cured for 10 seconds (Bluephase, Ivoclar Vivadent, 1000 mW/cm^2).

A split Teflon mold (diameter 3.6 mm; height 2.5 mm) was clamped to the treated dentin surface and filled with resin composite (Tetric EvoCeram A3, Ivoclar Vivadent; Filtek Supreme XT A3, 3M ESPE, St Paul, MN, USA). Continuous or pulse-delay curing was performed with one of four LED-curing units (Bluephase, Ivoclar Vivadent; LEDemetron 1, sds Kerr, Orange, CA, USA; DioPower, CMS–Dental ApS, Copenhagen, Denmark; Elipar Freelight, 3M ESPE) and is described in Table 1. The power densities emitted by the various curing units, verified by using a radiometer (LED Radiometer, Demetron, sds Kerr), were compensated for by the curing period to give an energy density totaling 16 J/cm^2. For the pulse-delay technique, two seconds of light curing was followed by a one-minute interval, after which light curing was completed. For each of the eight curing protocols, 16 specimens were produced with each resin composite.

Table 1: *Mean Shear Bond Strength (MPa) and Standard Deviation for Tetric EvoCeram and Filtek Supreme XT According to the Curing Mode and Mode of Aging*

	Curing Unit	Power Density	Time	Tetric No Thermocycling	Filtek No Thermocycling	Tetric Thermocycling	Filtek Thermocycling
Continuous	Bluephase	1000 mW/cm²	16 seconds	22.0 ± 2.8	24.4 ± 4.0	23.3 ± 4.4	29.8 ± 5.7
	LEDemetron 1	720 mW/cm²	22 seconds	19.2 ± 4.4	26.2 ± 4.3	24.8 ± 6.3	26.2 ± 7.9
	DioPower	550 mW/cm²	29 seconds	22.2 ± 5.5	22.8 ± 2.6	22.7 ± 6.1	27.5 ± 7.4
	Freelight	200 mW/cm²	80 seconds	20.4 ± 6.6	25.5 ± 4.4	21.0 ± 4.3	22.3 ± 6.3
Pulse-delay two seconds–one minute*	Bluephase	1000 mW/cm²	14 seconds	18.0 ± 5.7	24.0 ± 2.7	24.1 ± 6.4	24.4 ± 8.0
	LEDemetron 1	720 mW/cm²	20 seconds	19.5 ± 2.5	19.8 ± 4.3	21.9 ± 5.3	23.5 ± 5.4
	DioPower	550 mW/cm²	27 seconds	18.4 ± 4.2	21.7 ± 5.8	21.9 ± 8.0	25.5 ± 6.3
	Freelight	200 mW/cm²	78 seconds	16.0 ± 3.9	21.3 ± 7.1	25.0 ± 4.7	21.8 ± 5.5

**For the pulse-delay groups, two additional seconds should be added to the exposure durations in the time column, corresponding to the initial pulse activation.*

Table 2: *Results of the 4-factorial ANOVA: Dependent Variable: Shear Bond Strength*

Effect	df	ss	ms	F	p
Mode of Curing (A)	1	279.39	279.39	9.17	0.0027*
Power Density (B)	3	139.19	46.40	1.52	0.2093
Resin Composite (C)	1	539.23	539.23	17.70	<0.0001*
Mode of Aging (D)	1	489.29	489.29	16.06	<0.0001*
A*B	3	22.42	7.47	0.25	0.8646
A*C	1	35.37	35.37	1.16	0.2824
A*D	1	53.60	53.60	1.76	0.1860
B*C	3	24.56	8.20	0.27	0.8477
B*D	3	23.19	7.73	0.25	0.8586
C*D	1	46.58	46.58	1.53	0.2175
A*B*C	3	33.22	11.07	0.36	0.7795
A*B*D	3	96.08	32.03	1.05	0.3707
A*C*D	1	30.26	30.26	0.99	0.3200
B*C*D	3	146.84	48.95	1.61	0.1886
Error	227	6915.46	30.46		

**Significant effect*

Ten minutes after completion of the light curing, the bonded specimens were freed from their molds and stored in water at 37°C for seven days. The teeth were then equally divided into two groups. Half of the specimens were immediately submitted to shear bond strength testing. The other half was thermocycled for 6,000 cycles between 5°C and 55°C baths before shear strength testing. Shear bond strength testing was performed in a universal testing machine (Instron, High Wycombe, United Kingdom) at a crosshead speed of 1 mm/minute. Mean values and standard deviation were calculated from the eight specimens in each group.

Analysis of the Failure Modes

The failure modes were evaluated at 18x magnification under a stereomicroscope. Failure was characterized as primarily being adhesive (A), primarily being cohesive within dentin (CD), primarily being cohesive within resin composite (CR) or mixed (M).

Statistical Methods

Shear bond strength data were submitted to factorial analysis of variance. Failure mode data were grouped within curing mode, power density, resin composite material or mode of aging before testing for significant effects. A Chi-square test was used to test for the number of adhesive failures against the combination of the other types of failure. The level of significance was defined as α=0.05.

RESULTS

Table 1 shows the mean values and standard deviations of bond strength for the different conditions investigated.

Four factorial analysis of variance were used to investigate influence of the mode of curing (continuous or pulse-delay), power density, resin composite material (Tetric EvoCeram or Filtek Supreme XT) and mode of aging (thermocycling or not) on shear bond strength (Table 2). The mode of curing, resin composite and mode of aging significantly influenced shear bond strength to dentin, whereas, power density had no effect. No significant interaction effects could be evidenced.

Regarding the failure mode (Table 3), significant effects of the curing mode (p=0.0288) and resin composite material (p=0.0022) were detected: an increased number of adhesive failures were registered for pulse-delay curing when compared with continuous curing and for Tetric EvoCeram when compared with Filtek Supreme XT. Thermocycling (p=1) and power density (p=0.2261) did not affect the failure mode.

Table 3: *Number of Specimens Out of Eight That Showed Adhesive Failure*

	Curing Unit	Tetric No Thermocycling	Filtek No Thermocycling	Tetric Thermocycling	Filtek Thermocycling
Continuous	Bluephase	0	0	0	0
	LEDemetron 1	1	0	0	0
	DioPower	0	0	4	0
	Freelight	0	0	1	0
Pulse-delay two seconds–one minute*	Bluephase	2	0	1	0
	LEDemetron 1	1	1	3	1
	DioPower	3	0	1	1
	Freelight	2	1	0	0

DISCUSSION

Varying the rate of cure by varying the power density did not affect shear bond strength. This is apparently in contrast with previous works by Uno and Asmussen[25] and Miyasaki and others,[26] in which bond strength to dentin decreased as power density decreased. However, as the energy density was not kept constant in these studies and, in fact, it decreased along with power density, the decline in bond strength caused by reduced power density may have reflected a reduced degree of cure instead of a reduced rate of cure.

Varying the rate of cure by varying the curing mode significantly influenced shear bond strength, which leads to partial rejection of the null hypothesis. Pulse-delay curing resulted in lower shear bond strength of resin composite to dentin when compared to continuous curing. Although this was not expected, it is possible that the more linear polymer structure, previously reported to result from this curing mode,[22,24] affected the mechanical properties of resin composite materials to such an extent that it influenced bond strength. It has also been observed that, at a given energy density, pulse-delay curing may result in a lower degree of cure at 2-mm depths when compared to continuous irradiation.[27] This observation constitutes a further possible explanation for the findings in this study, including the observation that pulse-delay curing resulted in more adhesive failures than did continuous cure. The effect of curing mode on bond strength has been previously investigated. Earlier studies compared the effect of step-cure and continuous cure, with divergent results being observed.[17-18] In agreement with the results of the current study, Price and others[17] found that changing from continuous to step-cure reduced the bonding of resin composite to dentin. Caldwell, Kulkarni and Titley,[18] on the other hand, did not register significant differences between step-cure and continuous cure regarding bond strength. However, as opposed to the current work, there was no standardization of energy density emitted for the different groups, nor was the power density systematically varied in previous publications.

Filtek Supreme XT was found to give rise to higher bond strengths than Tetric EvoCeram. The mechanical properties of resin composites are reflected in bond strength.[28-30] Filtek Supreme XT has been found to have higher modulus of elasticity as compared to Tetric EvoCeram (Asmussen & Peutzfeldt, 2006, unpublished results), which may explain why Filtek Supreme resulted in significantly higher bond strengths and significantly fewer adhesive failures than Tetric EvoCeram.

Thermocycling increased the bonding of resin composite to dentin. It is possible that the increase in temperature caused by the hot bath (55°C) has improved the degree of conversion of the resin composite and that this was reflected in shear bond strength. Thus, when resin composite materials were exposed to heat after light irradiation,[31] an increase in the degree of conversion was observed. However, ranking of the groups was maintained after thermocycling and has been likewise observed in other publications.[20-21,32] Thermocycling may still be considered a valuable means to stress the adhesive interface.[19] It must be noted, however, that the influence of thermocycling on bond strength may depend on the specific test set-up or adhesive used.[20,32]

CONCLUSIONS

Compared to continuous curing, pulse-delay curing reduced the bonding of resin composite to dentin at a constant energy density. Therefore, when deciding on a specific curing protocol, clinicians are advised to be aware of the advantages and limitations of each curing mode.

Acknowledgements

The authors acknowledge support from CAPES (Coordenação de Aperfeiçoamento de Pessoal de Nível Superior), Brazil, process PDEE 1908/05-7.

(Received 6 March 2006)

References

1. Rueggeberg FA, Caughman WF & Curtis JW Jr (1994) Effect of light intensity and exposure duration on cure of resin composite *Operative Dentistry* **19(1)** 26-32.
2. Yap AU & Senevirante C (2001) Influence of light energy density on effectiveness of composite cure *Operative Dentistry* **26(5)** 460-466.
3. Halvorson RH, Erickson RL & Davidson CL (2002) Energy dependent polymerization of resin-based composite *Dental Materials* **18(6)** 463-469.
4. Emami N & Söderholm KJ (2003) How light irradiance and curing time affect monomer conversion in light-cured resin composites *European Journal of Oral Sciences* **111(6)** 536-542.
5. Peutzfeldt A & Asmussen E (2005) Resin composite properties and energy density of light cure *Journal of Dental Research* **84(7)** 659-662.
6. Lovell LG, Lu H, Elliott JE, Starsbury JW & Bowman CN (2001) The effect of cure rate on the mechanical properties of dental resins *Dental Materials* **17(6)** 504-511.
7. Asmussen E & Peutzfeldt A (2005) Polymerization contraction of resin composite vs energy and power density of light-cure *European Journal of Oral Sciences* **113(5)** 417-421.
8. Asmussen E & Peutzfeldt A (2001) Polymerization of resin composites and the influence on properties *Odontoiatria Adesiva e Ricostruttiva* **5** 55-61.
9. Braga RR & Ferracane JL (2002) Contraction stress related to degree of conversion and reaction kinetics *Journal of Dental Research* **81(2)** 114-118.
10. Davidson CL & Feilzer AJ (1997) Polymerization shrinkage and polymerization shrinkage stress in polymer-based restoratives *Journal of Dentistry* **25(6)** 435-440.
11. Uno S & Asmussen E (1991) Marginal adaptation of a restorative resin polymerized at reduced rate *Scandinavian Journal of Dental Research* **99(5)** 440-444.
12. Feilzer AJ, Dooren LH, de Gee AJ & Davidson CL (1995) Influence of light intensity on polymerization shrinkage and integrity of restoration-cavity interface *European Journal of Oral Sciences* **103(5)** 322-326.
13. Sahafi A, Peutzfeldt A & Asmussen E (2001) Effect of pulse-delay curing on *in vitro* wall-to-wall contraction of composite in dentin cavity preparations *American Journal of Dentistry* **14(5)** 295-296.
14. Kanca J 3rd & Suh BI (1999) Pulse activation: Reducing resin-based composite contraction stresses at the enamel cavosurface margins *American Journal of Dentistry* **12(3)** 107-112.
15. Chye CH, Yap AU, Laim YC & Soh MS (2005) Post-gel polymerization shrinkage associated with different light curing regimens *Operative Dentistry* **30(4)** 474-480.
16. Sakaguchi RL, Wiltbank BD & Murchison CF (2004) Contraction force rate of polymer composites is linearly correlated with irradiance *Dental Materials* **20(4)** 402-407.
17. Price RB, Bannerman RA, Rizkalla AS & Hall GC (2000) Effect of stepped vs continuous light curing exposure on bond strengths to dentin *American Journal of Dentistry* **13(3)** 123-128.
18. Caldwell R, Kulkarni G & Titley K (2001) Does single versus stepped curing of composite resins affect their shear bond strength? *Journal of the Canadian Dental Association* **67(10)** 588-592.
19. Ernst CP, Canbek K, Euler T & Willershausen B (2004) *In vivo* validation of the historical *in vitro* thermocycling temperature range for dental materials testing *Clinical Oral Investigations* **8(3)** 130-138.
20. Kerby RE, Knobloch LA, Clelland N, Lilley H & Seghi R (2005) Microtensile bond strengths of one-step and self-etching adhesive systems *Operative Dentistry* **30(2)** 195-200.
21. Huang MS, Li MT, Huang FM & Ding SJ (2004) The effect of thermocycling and dentine pre-treatment on the durability of the bond between composite resin and dentine *Journal of Oral Rehabilitation* **31(5)** 492-499.
22. Asmussen E & Peutzfeldt A (2001) Influence of pulse-delay curing on softening of polymer structures *Journal of Dental Research* **80(6)** 1570-1573.
23. Asmussen E & Peutzfeldt A (2003) Two-step curing: Influence on conversion and softening of a dental polymer *Dental Materials* **19(6)** 466-470.
24. Soh MS & Yap AU (2004) Influence of curing modes on crosslink density in polymer structures *Journal of Dentistry* **32(4)** 321-326.
25. Uno S & Asmussen E (1992) Selected variables in bonding to dentin *Scandinavian Journal of Dental Research* **100(2)** 130-132.
26. Miyazaki M, Hinoura K, Onose H & Moore BK (1995) Influence of light intensity on shear bond strength to dentin *American Journal of Dentistry* **8(5)** 245-248.
27. Hackman ST, Pohjola RM & Rueggeberg FA (2002) Depths of cure and effect of shade using pulse-delay and continuous exposure photo-curing techniques *Operative Dentistry* **27(6)** 593-599.
28. Zidan O, Asmussen E & Jorgensen KD (1980) Correlation between tensile and bond strength of composite resins *Scandinavian Journal of Dental Research* **88(4)** 348-351.
29. van Noort R, Noroozi S, Howard IC & Cardew G (1989) A critique of bond strength measurements *Journal of Dentistry* **17(2)** 61-67.
30. Yanagawa T & Finger WJ (1994) Relationship between degree of polymerization of resin composite and bond strength to Gluma-treated dentin *American Journal of Dentistry* **7(3)** 157-160.
31. Peutzfeldt A & Asmussen E (2000) The effect of postcuring on quantity of remaining double bonds, mechanical properties, and *in vitro* wear of two resin composites *Journal of Dentistry* **28(6)** 447-452.
32. De Munck J, Van Landuyt K, Coutinho E, Poitevin A, Peumans M, Lambrechts P & Van Meerbeek B (2005) Microtensile bond strength of adhesives bonded to Class I cavity-bottom dentin after thermocycling *Dental Materials* **21(11)** 999-1007.