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Effects of the Solvent Evaporation Technique on the Degree of Conversion of One-Bottle Adhesive Systems

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Clinical Relevance

Since methods of solvent evaporation can change the degree of conversion for some adhesives, practitioners should be aware of the type and composition of bonding agents used.

SUMMARY

This study evaluated the effect of four methods of solvent evaporation on the degree of conversion (DC) of seven one-bottle adhesive systems: Excite (EX), ONE-STEP (OS), Optibond Solo Plus (OB), Prime&Bond 2.1 (PB), Prime&Bond NT (NT), Single Bond (SB) and Single Bond Plus (SP) using Fourier Transform Infrared Analysis (FTIR). Adhesive resins were: 1) applied to KBr pellet surfaces and left undisturbed for 30 seconds (condition 1), 2) left undisturbed for 30 seconds and air-dried with an air stream for 10 seconds (condition 2), 3) left undisturbed for 60 seconds (condition 3) and 4) left undisturbed for 60 seconds and air-dried for 10 seconds (condition 4) before curing. FTIR spectra were obtained and the DC was calculated by comparing the ratio of aliphatic/aromatic double carbon bonds before and after light-activation for 10 seconds (XL 3000, 3M). The results of each product were analyzed by one-way repeated measure ANOVA and post-hoc Tukey's test (*p*<0.05). The DC of PB, NT, OB and SP adhesives was not affected by the four evaporation conditions, while the DC of EX, OS and SB changed according to the evaporation method. The results suggested that the DC of some adhesives was similar regardless of the evaporation method when no water from dentin or rinsing was involved. Other bonding agents

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showed higher DC after specific conditions of solvent evaporation.

INTRODUCTION

Some dental bonding agents are composed of a mixture of hydrophilic primers and hydrophobic adhesive resins in a single solution.[1] These adhesive systems require previous phosphoric acid-etching, rinsing with water to remove the acid gel and creating moist surfaces before the application of adhesive resin to the dentin substrate. The resin monomers are dissolved in organic solvents, such as acetone and ethanol, which improve monomer diffusion into the demineralized matrix and play an important role in the removal of water during solvent evaporation.[2-4]

The high quality of the hybridization process depends on optimal monomer infiltration between the collagen fibrils of the demineralized matrix and the removal of as much water and organic solvents as possible from the surface prior to curing.[5-6] Compressed air has been used to accelerate solvent and water evaporation, ensuring better conditions of resin polymerization, which relates to the durability of the resin-dentin bonds in the wet bonding technique.[2,7]

The use of an air spray to accelerate solvent evaporation has been recommended by the manufacturers, since the remaining high content of water and organic solvents can greatly inhibit the polymerization reaction and compromise the creation of a well-defined polymer matrix.[4-5,8] Thus, clinicians must be aware of the volatile characteristics and composition of adhesive systems to achieving effective bonding with one-bottle adhesive systems. This study evaluated the degree of conversion of seven adhesive systems photo-activated after four methods of solvent evaporation using Fourier Transform Infrared (FTIR) analysis. The null hypothesis was that there is no difference in the degree of conversion when adhesive systems are light-activated after different conditions of solvent evaporation.

METHODS AND MATERIALS

The adhesive systems (respective organic solvent) investigated were Excite (ethanol), ONE-STEP (acetone), Optibond Solo Plus (ethanol), Prime&Bond 2.1 (acetone), Prime&Bond NT (acetone), Single Bond (ethanol) and Single Bond Plus (ethanol). Their compositions and manufacturers are listed in Table 1. Each bonding agent was tested according to four conditions of solvent evaporation (n=5). The analyses were performed at 24°C, under 64% relative humidity.

For FTIR analysis of the bonding agents, one drop of adhesive resin was dispensed into a well immediately prior to application. An applicator tip was dipped into the adhesive solution and the adhesive was applied to the surface of a potassium bromide pellet (LabSynth, Diadema, SP, Brazil). Before curing, the adhesive resin was left undisturbed on the pellet surface for 30 seconds, 60 seconds, 30 seconds and air-dried with a mild oil-free air stream for 10 seconds (40 pounds and a distance of 10 mm between the pellet surface and tip of an air-syringe) or it was left undisturbed for 60 seconds and air-dried with a mild oil-free air stream for 10 seconds. An FTIR spectrum of unpolymerized adhesive solution was obtained from each sample using 20 scans at 4 cm^{-1} in the transmittance mode (Equinox 55, Bruker Optik GmbH, Ettlingen, Germany).

Table 1: *Composition, Manufacturer and Lot Number of the Adhesive Systems Used in This Study*

Adhesive Systems	Composition	Manufacturer	Lot #
Excite	HEMA, phosphonic acid acrylate, silicon dioxide. Solvent: ethanol	Ivoclar Vivadent, Schan, Liechtenstein	F68154
ONE-STEP	BPDM, HEMA, Bis-GMA Solvent: acetone	BISCO Inc, Schuamburg, IL, USA	0400007894
Optibond Solo Plus	Alkyl dimethacrylate resins, barium aluminoborosilicate glass, silicon dioxide, sodium hexafluorosilicate Solvent: ethanol	Kerr Corp, Orange, CA, USA	405282
Prime&Bond 2.1	PENTA, dimethacrylate resins, urethane di-methacrylate resin, cetylamine hydrofluoride Solvent: acetone	Dentsply DeTrey, Konstanz, Germany	307244
Prime&Bond NT	PENTA, di- and tri-methacrylate resins, cetylamine hydrofluoride, urethane di-methacrylate resin, silica colloidal Solvent: acetone	Dentsply DeTrey, Konstanz, Germany	168410
Adper Single Bond	HEMA, Bis-GMA, water, PAA Solvent: ethanol	3M ESPE, St Paul, MN, USA	4JU
Adper Single Bond Plus	HEMA, Bis-GMA, water, PAA, coloidal silica Solvent: ethanol	3M ESPE, St Paul, MN, USA	4BR

Abreviations: HEMA: 2-hydroxyethyl methacrylate; BPDM: biphenyl dimethacrylate; Bis-GMA: bisphenol A diglycidyl ether dimethacrylate; PENTA: dipentaerythritol penta-acrylate monophosphate PAA: methacrylate functional copolymer of acrylic and polyitaconic acids.

The adhesive systems were light cured for 10 seconds (600 mW/cm^2) using a conventional quartz-tungsten-halogen light source (XL 3000, 3M ESPE, St Paul, MN, USA). The tip of the curing unit was positioned 3 mm from the pellet surface. Additional FTIR spectra were obtained immediately after solvent evaporation methods and light curing.

For analysis of the degree of conversion, the aliphatic carbon-to-carbon double bond absorbance peak intensity, located at 1638 cm^{-1}, and that for the aromatic component, located at 1608 cm^{-1} (aromatic), were compared in each spectrum before and after the polymerization reaction, and monomer conversion was determined using the following equation[9]:

$$(\%\ C{=}C)\ \frac{[\text{abs (aliphatic C=C)/abs (aromatic C...C)] polymer}}{[\text{abs (aliphatic C=C)/abs (aromatic C...C)] monomer}} \times 100$$

(% C=C): Percentage of remaining carbon double bonds.
abs : absorbance

The degree of conversion was obtained by subtracting the percentage of the remaining carbon double bonds (% C=C) from 100%. Only intra-brand conversion values were compared by one-way repeated measure Analysis of Variance and Tukey's post hoc test. Statistical significance was established at $\alpha=0.05$.

RESULTS

Mean monomer conversion values are presented in Table 2. The degree of conversion of Optibond Solo Plus, Prime&Bond 2.1, Prime&Bond NT and Single Bond Plus adhesive systems was not affected by the different conditions of solvent evaporation ($p>0.05$). The application of these adhesives to potassium bromide pellets and light-activation after 30 seconds without air-drying promoted a similar degree of conversion when compared with other treatments.

Excite adhesive did not require an air-drying step to reach a higher degree of conversion, while the ONE-STEP and Single Bond systems presented a higher degree of conversion after the air-drying step to evaporate the solvents. For these tested materials, monomer conversion values were wide ranging, according to variation of the technique of solvent evaporation used ($p<0.05$).

Figures 1 and 2 show the spectrum sites obtained from the ONE-STEP and Prime&Bond NT adhesive systems before (uncured state) and immediately after light curing. Figures 1A and 2A represent the spectra obtained when adhesive resins were left undisturbed on the pellet surface for 30 seconds, while Figures 1B and 2B represent the spectra for the same treatment, but when the adhesives were air-dried with an air stream for 10 seconds before curing. For the Prime&Bond NT adhesive, the height of peaks located at 1639 cm^{-1} after light curing were similar for the different evaporation conditions (Figures 2A and 2B). Compared to the ONE-STEP spectra obtained from the air-dried samples (Figure 1B), the spectra obtained from samples without air-drying demonstrated a slight

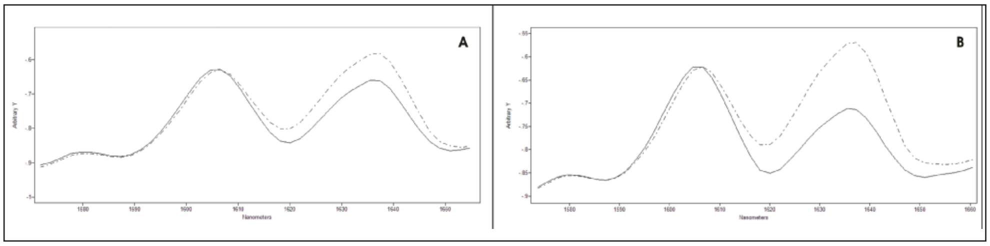

Figure 1: *FTIR spectrum sites obtained from ONE-STEP adhesive before light exposure (segmented line) and after curing. A) adhesive resin was applied to the KBr pellet surface and were left undisturbed for 30 seconds before light curing for 10 seconds. B) adhesive resin was applied to the KBr pellet surface, left undisturbed for 30 seconds, air-dried for 10 seconds and light-cured for 10 seconds.*

Table 2: *Degree of Conversion Means (± SD) of Adhesive Systems (organic solvent) According to Evaporation Conditions of Solvents*

Experimental Groups	Excite (ethanol)	ONE-STEP (acetone)	Optibond Solo Plus (ethanol)	Prime&Bond 2.1 (acetone)	Prime&Bond NT (acetone)	Single Bond (ethanol)	Single Bond Plus (ethanol)
Light-activation after 30 seconds	33.5±4.1 AB	37.2±1.2 A	45.4±1.0 A	45.3±6.3 A	35.1±1.2 A	24.1±2.6 A	19.4±3.7 A
Light-activation after 60 seconds	37.5±1.2 A	37.1±2.8 A	43.4±1.5 A	44.1±7.2 A	35.3±2.4 A	24.6±3.0 A	19.3±3.5 A
Light-activation after 30 seconds and air-drying (10 seconds)	28.4±1.2 B	44.4±4.7 B	43.6±3.6 A	40.3±13.0 A	30.6±8.5 A	27.2±3.5 AB	21.7±4.6 A
Light-activation after 60 seconds and air-drying (10 seconds)	32.4±4.2 AB	46.4±3.7 B	43.0±2.5 A	43.9±11.5 A	30.5±4.9 A	30.7±2.1 B	21.7±4.4 A

Means followed by different letters (capital letter-column) differ statistically by Tukey test (p<0.05). No comparison among the products was performed.

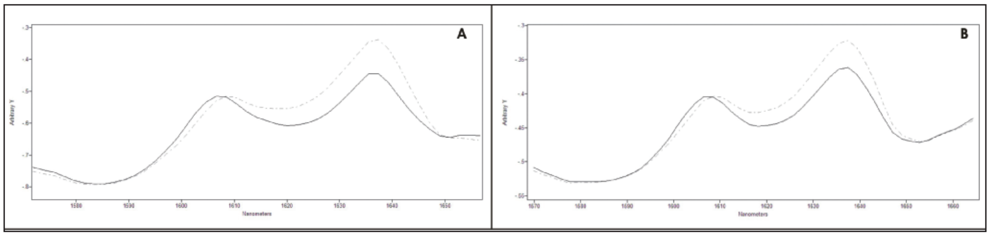

Figure 2: *FTIR spectrum sites obtained from Prime&Bond NT adhesive before light exposure (segmented line) and after curing. A) adhesive resin was applied to the KBr pellet surface and left undisturbed for 30 seconds before light curing for 10 seconds. B) adhesive resin was applied to the KBr pellet surface, left undisturbed for 30 seconds, air-dried for 10 seconds and light-cured for 10 seconds.*

reduction in the peak, corresponding to aliphatic carbon-double bonds (1639 cm^{-1}) (Figure 1A).

DISCUSSION

The results of this study showed that the degree of conversion of bonding agents did not depend solely on the type of organic solvent; instead, it depended on the monomeric composition contained in the organic solvent solution (ethanol or acetone) of each material. The monomer conversion of two ethanol-based (Optibond Solo Plus and Single Bond Plus) and two acetone-based (Prime&Bond 2.1 and NT) adhesives was not influenced by evaporation methods. On the other hand, the other adhesives (Excite, ONE-STEP and Single Bond) showed changes in the degree of conversion as a function of the evaporation methods regardless of the type of organic solvents in their composition. Thus, the anticipated null hypothesis of the current study was only partially confirmed.

For ONE-STEP and Single Bond adhesives, the use of an air spray for 10 seconds to accelerate solvent evaporation was important for increasing monomer conversion. Studies have shown the difficulty in removing water in aqueous solutions of HEMA, which is the functional monomer of ONE-STEP and Single Bond systems.[10-11] Thus, air-drying is essential for providing higher degree of conversion values. After application, proper air-drying of the adhesive solution reduces water content and leaves only the priming resin at the dentin surface, resulting in a higher degree of conversion. Conversely, if a high content of organic solvents and water from adhesive composition remains, polymerization will be inhibited, compromising the quality of the hybrid layer.[5,10-11]

Excite, Optibond Solo Plus, Single Bond Plus, Prime&Bond 2.1 and Prime&Bond NT adhesives did not require air-drying to improve the degree of conversion, representing less sensitive materials for clinical use. The simple application of these adhesives to a potassium bromide pellet without disturbing the pellet for 30 seconds and allowing the pellet to dry at 24°C under 64% relative humidity showed a similar degree of conversion when compared with other conditions of solvent evaporation in which the increase in undisturbed time and air-drying were performed.

The adhesive systems showed a low degree of conversion due to the interference of atmospheric oxygen with adhesive resin polymerization.[12-13] However, a low degree of conversion can occur in clinical procedures once the adhesive systems are spread into a thin layer after application to minimize pooling and evaporate the solvent carrier. The low monomer conversion observed due to the presence of residual oxygen may result in an impaired hybrid layer formation and a greater chance for chemical incompatibility to occur between the bonding agents and restorative composites.[14-15]

Solvent evaporation can facilitate the polymerization reaction, because solvent volatilization can reduce the distance among monomers and increase the degree of conversion.[5,10-11,16] Conversely, the presence of filler particles increases adhesive resin viscosity, decreasing monomer conversion.[17-18] Therefore, the changes in composition caused by solvent and water removal in filled adhesive systems did not increase the degree of conversion compromised by the presence of fillers.

The presence of organic solvents in the efficacy of dentin bonding has been recognized.[4] Acetone is frequently used as a solvent, since it can efficiently remove water from surfaces.[3,5,19] By adding 10% acetone to water, vapor-pressure increases more than 300%, leading to volatilization of some of the surface water.[20] Ethanol is another organic solvent that is used as a vehicle in adhesives, but it presents a higher boiling temperature and less high vapor pressure than acetone. Alcohol triggers more hydrogen bonding than acetone. Thus, alcohol does not chase water as effectively as acetone, so that, consequently, the durability of dentin bonding can be compromised.[3,19,21] Conversely, acetone does not expand dried demineralized dentin as alcohol does and it must be used only with the wet-bonding technique.[8,22-25]

Although all manufacturers indicate the drying step after applying bonding agents to evaporate water and solvents from the adhesive solution, some products tested in this study did not require the drying step. However, no residual moisture from the wet bonding technique was mixed with adhesive solutions, which could completely alter the polymerization process and monomer conversion.[5,16] Since only the effects of adhesive composition in the evaluation of the degree of conversion after the evaporation of solvents was evaluated, the results observed in this study should not be extrapolated to the clinical situation where water is involved with bonding procedures.

Some bonding agents require an additional application of adhesive resin over the first light-activated adhesive resin layer. A proper monomer conversion of the second adhesive layer results in a thick, uniform layer beneath the resin composite. Since the adhesive layer consists of a low modulus resin material, a relatively thick (50-150 µm) adhesive layer has proven to be effective in leveling the mismatch of modulus values at the restoration-tooth structure interface.[26-28] Consequently, the maximum stress area at the bottom of the restoration decreases in the presence of a thick adhesive resin layer because of its elastic release effect.[29] Therefore, the results of the current study can be valuable in determining the best clinical procedure for different adhesive systems achieving proper monomer conversion and, consequently, creating a thick adhesive layer when the second adhesive layer is applied to dentin or when adhesive systems are used to bond composite to composite when the repair of composite restorations is required or when adhesives are used to bond composites to conditioned and dried enamel.

Dentists must follow the manufacturer's instructions when applying adhesive systems to wet dentin, because further research is necessary to explain the effects of mixing water and an adhesive solution on bonding to demineralized dentin. Moreover, studies have shown that solvents and adhesive monomers are responsible for changes in dimensional and other mechanical properties of the demineralized dentin matrix that compromises the hybridization process.[22-25,30] The degree of conversion of bonding agents is an important parameter to be evaluated, since high monomer conversion is related to a well-defined polymer matrix developed at the hybrid layer and to proper mechanical properties for long-lasting restorations.[16,31]

CONCLUSIONS

Within the limitations of this study and when no residual water from wet dentin or rinsing is involved in bonding procedures, for some adhesive systems, an inadequate drying step can interfere with polymerization, reducing monomer conversion. For other bonding agents, the types of solvents and evaporation conditions did not affect monomer conversion.

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References

1. Perdigão J, Ramos JC & Lambrechts P (1997) *In vitro* interfacial relationship between human dentin and one-bottle dental adhesives *Dental Materials* **13(4)** 218-227.
2. Pashley DH & Carvalho RM (1997) Dentine permeability and dentine adhesion *Journal of Dentistry* **25(5)** 355-372.
3. Abate PF, Rodriguez VI & Macchi RL (2000) Evaporation of solvent in one-bottle adhesives *Journal of Dentistry* **28(6)** 437-440.
4. Reis AF, Oliveira MT, Giannini M, de Goes MF & Rueggeberg FA (2003) The effect of organic solvents on one-bottle adhesives' bond strength to enamel and dentin *Operative Dentistry* **28(6)** 700-706.
5. Jacobsen T & Soderholm KJ (1995) Some effects of water on dentin bonding *Dental Materials* **11(2)** 132-136.
6. De Munck J, Van Landuyt K, Peumans M, Poitevin A, Lambrechts P, Braem M & Van Meerbeek B (2005) A critical review of the durability of adhesion to tooth tissue: Methods and results *Journal of Dental Research* **84(2)** 118-132.
7. Ikeda T, De Munck J, Shirai K, Hikita K, Inoue S, Sano H, Lambrechts P & Van Meerbeek B (2005) Effect of evaporation of primer components on ultimate tensile strengths of primer-adhesive mixture *Dental Materials* **21(11)** 1051-1058.
8. Carvalho RM, Mendonca JS, Santiago SL, Silveira RR, Garcia FC, Tay FR & Pashley DH (2003) Effects of HEMA/solvent combinations on bond strength to dentin *Journal of Dental Research* **82(8)** 597-601.
9. Ferracane JL & Greener EH (1984) Fourier transform infrared analysis of degree of polymerization in unfilled resins—methods comparison *Journal of Dental Research* **63(8)** 1093-1095.
10. Pashley EL, Zhang Y, Lockwood, PE, Rueggeberg FA & Pashley DH (1998) Effects of HEMA on water evaporation from water-HEMA mixtures *Dental Materials* **14(1)** 6-10.
11. Paul SJ, Leach M, Rueggeberg FA & Pashley DH (1999) Effect of water content on the physical properties of model dentine primer and bonding resins *Journal of Dentistry* **27(3)** 209-214.
12. Ruyter IE (1981) Unpolymerized surface layers on sealants *Acta Odontologica Scandinavica* **39(1)** 27-32.
13. Rueggeberg FA & Margeson DH (1990) The effect of oxygen inhibition on an unfilled/filled composite system *Journal of Dental Research* **69(10)** 1652-1658.
14. Sanares AME, Itthagarun A, King NM, Tay FR & Pashley DH (2001) Adverse surface interactions between one-bottle light-cured adhesives and chemical-cured composites *Dental Materials* **17(6)** 542-556.
15. Giannini M, de Goes MF, Nikaido T, Shimada Y & Tagami J (2004) Influence of activation mode of dual-cured resin composite cores and low-viscosity composite liners on bond strength to dentin treated with self-etching adhesives *Journal of Adhesive Dentistry* **6(4)** 301-306.
16. Nunes TG, Garcia FCP, Osorio R, Carvalho RM & Toledano M (2006) Polymerization efficacy of simplified adhesive systems studied by NMR and MRI techniques *Dental Materials* **22(10)** 963-972.

17. Ferracane JL, Berge HX & Condon JR (1998) *In vitro* aging of dental composite in water-effect of degree of conversion, filler volume, and filler/matrix coupling *Journal of Biomedical Materials Research* **42(3)** 465-472.

18. Giannini M, Arrais CAG & Rueggeberg FA (2006) Effect of filler particles on degree of conversion of one-bottle adhesive systems *Brazilian Oral Research* **20(Supplement)** Abstract #Pb279 p 244.

19. Kanca J III (1992) Effect of resin primer solvents and surface wetness on resin composite bond strength to dentin *American Journal of Dentistry* **5(4)** 213-215.

20. Chaudhry MM, Van Ness HC & Abbott MM (1980) Excess thermodynamic functions for ternary systems. 6. Total-pressure data and GE for acetone-ethanol-water at 500C *Journal of Chemical & Engineering Data* **25(3)** 254-257.

21. Jacobsen T & Soderholm KJM (1998) Effect of primer solvent, primer agitation, and dentin dryness on shear bond strength to dentin *American Journal of Dentistry* **11(5)** 225-228.

22. Maciel KT, Carvalho RM, Ringle RD, Preston CD, Russell CM & Pashley DH (1996) The effects of acetone, ethanol, HEMA, and air on the stiffness of human decalcified dentin matrix *Journal of Dental Research* **75(11)** 1851-1858.

23. Pashley DH, Carvalho RM, Tay FR, Agee KA & Lee KW (2002) Solvation of dried dentin matrix by water and other polar solvents *American Journal of Dentistry* **15(2)** 97-102.

24. Eddleston CL, Hindle AR, Agee KA, Carvalho RM, Tay FR, Rueggeberg FA & Pashley DH (2003) Dimensional changes in acid-demineralized dentin matrices following the use of HEMA-water versus HEMA-alcohol primers *Journal of Biomedical Material Research* **Part A 67(3)** 900-907.

25. Pashley DH, Agee KA, Carvalho RM, Lee WK, Tay FR & Callison TE (2003) Effects of water-free polar solvents on the tensile properties of demineralized dentin *Dental Materials* **19(5)** 347-352.

26. Magne P, Versluis A & Douglas WH (1999) Effect of luting composite shrinkage and thermal loads on the stress distribution in porcelain laminate veneers *Journal of Prosthetic Dentistry* **81(3)** 335-344.

27. Davdison CL & Davidson-Kaban SS (1998) Handling of mechanical stresses in composite restorations *Dental Update* **25(7)** 274-279.

28. Ausiello P, Rengo S, Aspicella A & Davidson CL (2001) 3D-finite element analyses of cusp movements in a human upper premolar, restored with resin-based composites *Journal of Biomechanics* **34(10)** 1269-1277.

29. Ausiello P, Aspicella A & Davidson CL (2002) Effect of adhesive layer on stress distribution in composite restorations—a 3D finite element analysis *Dental Materials* **18(4)** 295-303.

30. Pashley DH, Agee KA, Nakajima M, Tay FR, Carvalho RM, Terada RSS, Harmon FJ, Lee WK & Rueggeberg FA (2001) Solvent-induced dimensional changes in EDTA-demineralized dentin matrix *Journal of Biomedical Material Research* **56(2)** 273-281.

31. Arrais CAG, Giannini M & Rueggeberg FA (2006) Monomer conversion of dual-cured adhesive systems: Effect of curing conditions *Journal of Dental Research* **85(Special Issue A)** Abstract #0294 p 294.