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Degree of Conversion of Simplified Contemporary Adhesive Systems as Influenced by Extended Air-Activated or Passive Solvent Volatilization Modes

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Clinical Relevance

Active air-drying for 60 seconds to volatilize solvents can be necessary to increase the degree of conversion of some adhesive systems, which might be related to improved clinical performance.

SUMMARY

This study evaluated the effect of five methods of solvent volatilization on the degree of conversion (DC) of nine one-bottle adhesive systems using Fourier transform infrared/attenuated total reflectance (FTIR/ATR) analysis. Nine adhesives were tested: Adper Single

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Bond 2 (SB), Adper Easy One (EO), One Up Bond F Plus (OUP), One Coat Bond SL (OC), XP Bond (XP), Ambar (AM), Natural Bond (NB), GO, and Stae. The adhesive systems were applied to a zinc-selenide pellet and 1) cured without solvent volatilization, 2) left undisturbed for 10 seconds before curing, 3) left undisturbed for 60 seconds before curing, 4) air-dried with an air stream for 10 seconds before curing, and 5) air-dried with an air stream for 60 seconds before curing. FTIR/ATR spectra were obtained, and the DC was calculated by comparing the aliphatic bonds/reference peaks before and after light activation for 10 seconds (FlashLite 1401). The DC means of each material were analyzed by one-way analysis of variance and post hoc Tukey test ($p<0.05$). The DC of GO and Stae adhesive systems was not affected by the five evaporation conditions. Air-drying for 60 seconds before curing yielded the highest DC for SB, EO, and OC. Extended solvent volatilization time (60 seconds) either with or without air-drying before curing provided the highest DC for AM, NB, XP, and OUP. Thus, the monomer conversion of adhesive systems was material dependent. In general, the 60-second passive or active air-drying modes to volatilize solvents before curing enhanced the degree of conversion for the one-bottle simplified adhesive systems.

INTRODUCTION

The development of adhesive systems has completely changed the traditional concepts of dentistry. Today, adhesive systems are widely used in direct procedures for restoration of anterior and posterior cavities, fissure sealing, reattachment of fractured fragments, and corrections in tooth morphology and in indirect procedures involving cementation of root-canal posts and indirect ceramic and composite crowns.[1]

Based on the management of the smear layer substrate, contemporary adhesive systems are categorized as etch-and-rinse (ER) and self-etch (SE) systems. Both bonding strategies are also available in a full or simplified version. When the conditioning step is followed by a priming step and application of the adhesive resin, ER adhesives are available in three steps, or they are available in a two-step procedure when the primer and adhesive resin are joined into one application. Similarly, SE adhesives can employ two steps or a single one, depending on the way the acidic primer and bonding resin are provided by the manufacturer.[2] An immediate consequence of adhesive simplification is sacrifice of the universality of multistep adhesives,[3] and simplified systems are currently preferred by clinicians to perform adhesive procedures because of time savings.

Successful adhesion to hard tissues is a fundamental requirement prior to the placement of dental materials and is directly dependent on the quality of the hybrid layer. Hence, any approach to prolong the lifetime of adhesives might focus on improving the stability of the bonding interface of these biomaterials to tooth tissues.[4] Optimal monomer infiltration into the demineralized substrates and the achievement of a high degree of conversion (DC) are crucial factors in establishing long-lasting bonds.[5] A low DC of dental adhesives is associated with low bond strength values and mechanical properties, high monomer elution, increased permeability, and phase separation.[6-8] Moreover, reduced DC even accounts for the possible continuous etching of the tooth substrate due to suboptimally polymerized acidic monomer in self-etch adhesives.[9] Thus, obtaining a high DC of adhesive systems is a crucial factor in the long-term stability of the hybrid layer.

Simplified dental bonding agents are composed of a mixture of hydrophilic primers and hydrophobic adhesive resins dissolved in acetone, ethanol, water, or some combination of the solvents, which play an important role in the bond performance.[10] Although the presence of solvents makes the process of monomer infiltration easier, the remaining water and organic solvents can greatly inhibit the polymerization reaction and compromise the creation of a well-defined polymer matrix.[11] The use of air spray to accelerate solvent evaporation has been recommended by the manufacturers, and several techniques have been evaluated by researchers. It has been shown that the extended passive solvent volatilization that occurs when adhesive systems are left undisturbed for 60 seconds with or without posterior air-drying for 10 seconds before photoactivation may increase the DC of some commercially available adhesive systems.[12] However, little is known about the DC of contemporary adhesive systems with respect to whether an extended air-activated drying mode to volatilize the solvent should be performed before curing. Thus, this study aimed to evaluate the DC of commercially available adhesive systems when photo-activated after extended air-activated or passive methods of solvent volatilization. The null hypothesis was that there is no difference in the DC of adhesive systems when photo-activated after different conditions of solvent evaporation.

MATERIALS AND METHODS

Experimental Design

Nine one-bottle commercially available adhesive systems with different solvents were tested: Adper Single Bond 2 (SB; 3M ESPE, St Paul, MN, USA), Adper Easy One (EO; 3M ESPE), One Up Bond F Plus (OUP; Tokuyama, Tokyo, Japan), One Coat Bond SL (OC; Coltène/Whaledent, Altstätten, Switzerland), XP Bond (XP; Dentsply/Caulk, Milford, DE, USA), Ambar (AM; FGM, Joinville, SC, Brazil), Natural Bond (NB; DFL, Rio de Janeiro, RJ, Brazil), GO (SDI, Victoria, Australia), and Stae (SDI). Moreover, five modes of solvent volatilization were performed before curing: 1) immediate cure without solvent volatilization, 2) passive solvent volatilization (left undisturbed) for 10 seconds, 3) passive solvent volatilization for 60 seconds (left undisturbed), 4) active solvent volatilization for 10 seconds (with air stream), and 5) active solvent volatilization for 60 seconds (with air stream). The composition, classification, manufacturers, and lot number of all adhesives systems tested are displayed in Table 1.

DC Analysis

The DC was analyzed by Fourier transform infrared/attenuated total reflectance (Spectrum 100, PerkinElmer, Shelton, XX, USA) at 24°C under 64% relative humidity. One drop of each adhesive system (n=5) was applied to the surface of a zinc selenide pellet (PerkinElmer). Before curing for 10 seconds with an LED light (FlashLite 1401, Discus Dental, Culver City, CA, USA; irradiance at 1100 mW/cm^2) positioned 3 mm from the pellet surface, the solvent of each adhesive resin was volatilized in accordance with the aforementioned modes.

The absorption spectra of nonpolymerized and polymerized adhesive resins were obtained from the region between 4000 and 650 cm^{-1} with 32 scans at 4 cm^{-1}. For adhesive systems containing aromatic vinyl bonds of bisphenol and aliphatic bonds of the methacrylate functional group (SB, EO, OC, OUP, AM, XP), the aliphatic carbon-to-carbon double-bond absorbance peak intensity (located at 1638 cm^{-1}) and that of the aromatic component (located at 1608 cm^{-1}; reference peak) were obtained. For Stae and GO, which do not present aromatic dimethacrylates, the intensity of the urethane reference peak (located at 1538 cm^{-1}) was obtained. For NB (a TEGDMA-based adhesive), the intensity of the carbonyl reference peak (located at 1716 cm^{-1}) was obtained. The DC (%) was calculated using the following equation: DC (%) = 100 × [1 − (R polymerized/R nonpolymerized)], where R represents the ratio between the absorbance peak at 1638 cm^{-1} and 1608 cm^{-1} (for SB, EO, OC, OUP, AM, and XP), 1638 cm^{-1} and 1537 cm^{-1} (for GO and Stae), and 1638 cm^{-1} and 1716 cm^{-1} (for NB).

The data were analyzed by one-way analysis of variance and post hoc Tukey test (only an intrabrand comparison was performed to compare the difference among the tested solvent volatilization modes for each adhesive resin). Statistical significance was established at α=0.05.

RESULTS

The means and standard deviations of the degree of conversion values are presented in Table 2. Only the application of air for 60 seconds yielded a statistically higher degree of conversion for SB, EO, and OC. Both extended times of solvent volatilization (60 seconds; active and passive methods) promoted statistically increased monomer conversion for AM, NB, XP, and OUP. On the other hand, the degree of conversion of GO and Stae was not influenced by the volatilization technique.

DISCUSSION

The null hypothesis tested in this study was rejected because the degree of conversion of most adhesives was affected by different solvent volatilization methods. Air volatilization for 60 seconds provided the statistically highest monomer conversion for two ethanol-based (SB and EO) and one water-based (OC) adhesive system tested, whereas either air or passive volatilization methods for 60 seconds yielded the highest monomer conversion for other ethanol-based (AM, NB) and water-based (OUP) adhesive systems and for the tertiary-alcohol-based (XP) adhesive system. Nevertheless, none of the tested solvent volatilization techniques affected the monomer conversion of the acetone-based adhesive systems evaluated in the present investigation.

Solvent volatilization can facilitate the polymerization reaction because the distance among monomers is reduced, increasing the degree of conversion.[13] Ideally, solvents should be completely volatilized from the applied mixture prior to polymerization. However, it has been shown that solvents cannot be completely removed from adhesive systems.[14] As water/solvent volatilizes from the

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

Adhesive Systems	Composition (% by Weight)	Classification	Manufacturer	Lot No.
Adper Single Bond 2	Ethyl alcohol (25-30), silane treated silica (nanofiller) (10-20), Bis-GMA (10-20), HEMA (5-10), glycerol 1,3-dimethacrylate (5-10), copolymer of acrylic and itaconic acids (5-10), water (<5), diurethane dimethacrylate (1-5)	Two-step E&R	3M ESPE, St Paul, MN, USA	8PT
Adper Easy One	Bis-GMA (15-25), HEMA (15-25), ethanol (10-15), water (10-15), phosphoric acid-6-methacryloxy-hexylesters (5-15), silane treated silica (8-12), 1,6-hexanediol dimethacrylate (5-10), copolymer of acrylic and itaconic acid (1-5), (dimethylamino)ethyl methacrylate (1-5), camphorquinone (1-3), 2,4,6-trimethylbenzoyldiphenylphosphine oxide (1-3)	One-step SE	3M ESPE, St Paul, MN, USA	84020
One Up Bond F Plus	Agent A: Methacryloyloxyalkyl acid phosphate (30-60), MAC-10 (10-30), methyl methacrylate (10-20), Bisphenol A polyethoxy methacrylate (20-40) Agent B: HEMA (30-60), methyl methacrylate (10-30), fluoroaminosilicate glass filler (10-15), borate catalyst (<5), purified water (5-20)	One-step SE	Tukoyama, Tokyo, Japan	61184
One Coat Bond SL	Methacrylate ([a]), polyalkenoat methacryliert ([a]), water ([a])	Two-step E&R	Coltène/Whaledent, Altsätten, Switerland	0173809
XP Bond	Methacrylates (25-50), tert-butyl alcohol (10-25), acrylates (10-25)	Two-step E&R	Dentsply/Caulk, Milford, DE, USA	17056CB
Ambar	UDMA (5-40), HEMA (5-40), methacrylate acidic monomers (1-20), methacrylate hydrophilic monomers (5-40), silanized silicon dioxide (<1), camphorquinone (<1), 4-EDAMB (<1), ethanol (<20)	Two-step E&R	FGM, Joinville, SC, Brazil	161210
Natural Bond	PMGDM ([a]), TEGDMA ([a]), HEMA ([a]), PHFA ([a]), camphorquinone ([a]), 4-EDAMB ([a]), butyl-hydroxytoluene ([a]), ethanol ([a])	Two-step E&R	DFL, Rio de Janeiro, RJ, Brazil	10121648
GO	Acetone (30-50), acrylic monomer (30-50), balance ingredient (non-hazardous) (10-15)	One-step SE	SDI, Victoria, Australia	164413
Stae	Acetone (54), acrylic monomer (44), balance ingredient (non-hazardous) (2)	Two-step E&R	SDI, Victoria, Australia	090301

Abbreviations: Bis-GMA, bisphenol A-glycidyl methacrylate; E&R, etch-and-rinse; EDAMB, ethyl 4-dimethylaminobenzoate; HEMA, 2-hydroxyethyl methacrylate; MAC-10, 11-methacryloxy-1,1-undecanedicarboxylic acid; PHFA, potassium hexafluoroantimonate; PMGDM, pyromellitic glycerol dimethacrylate; SE, self-etching; TEGDMA, triethyleneglycol dimethacrylate; UDMA, urethane dimethacrylate.

[a] *Not provided by the manufacturer.*

Table 2: *Degree of Conversion Means (Standard Deviations) of Adhesive Systems According to Volatilization Conditions of Solvents*[a]

Adhesive Systems	Immediate	10-Second Passive	60-Second Passive	10-Second Active	60-Second Active
Adper Single Bond 2	75.5 (0.3) C	75.0 (0.6) C	77.4 (0.7) B	77.1 (0.7) B	79.7 (0.2) A
Adper Easy One	48.9 (0.4) B	48.9 (0.2) B	49.6 (0.4) B	50.8 (0.1) B	55.5 (1.5) A
One Up Bond F Plus	89.9 (0.5) B	89.5 (0.7) B	94.6 (0.4) A	90.4 (0.3) B	95.7 (0.2) A
One Coat Bond SL	77.5 (0.3) C	78.5 (0.5) BC	80.1 (1.0) AB	79.5 (0.4) BC	82.2 (0.9) A
XP Bond	52.4 (0.3) B	58.1 (0.7) B	67.4 (0.8) A	66.0 (0.5) AB	68.8 (0.1) A
Ambar	39.8 (1.7) C	44.1 (2.0) BC	57.6 (4.8) A	50.4 (2.2) AB	58.2 (3.0) A
Natural Bond	24.3 (2.0) C	28.9 (3.8) C	51.6 (1.2) A	42.6 (2.5) B	50.8 (0.6) A
GO	82.9 (0.5) A	82.6 (0.6) A	83.7 (0.2) A	82.6 (0.8) A	83.9 (0.3) A
Stae	80.8 (0.1) A	80.8 (0.1) A	75.6 (0.9) A	79.1 (0.1) A	79.0 (0.4) A

[a] *Means followed by different capital letters differ statistically by Tukey test ($p \leq 0.05$). No comparison among the products was performed.*

adhesive, monomer density is found to increase sharply, creating a monomer concentration gradient that acts as a barrier for further solvent evaporation, reducing the ability of water and solvents to volatilize from the adhesive.[15] Thus, clinicians should attempt to remove the highest amount of solvent to achieve an adequate monomer conversion. In fact, a low DC of adhesive systems is associated with low bond strength values and mechanical properties, high monomer elution, increased permeability, and phase separation.[6-8] Moreover, reduced DC even accounts for the possible continuous etching of the tooth substrate due to suboptimally polymerized acidic monomer in self-etch adhesives.[9]

Several factors have been related to the solvent retention in adhesive systems. Solvents with relatively low vapor pressure, such as water, when mixed with nonvolatile monomers, become less able to volatilize as monomer concentration increases.[14] On the other hand, acetone, with a relatively high vapor pressure of 184 mm Hg at 20°C, volatilizes much faster than ethanol or water, with vapor pressure of 43.9 and 17.5 mm Hg, respectively.[16] Also, the extent of solvent retention in polymer networks depends on the resin polarity. The resin polarity influences the number of hydrogen bonding sites and the attraction between the polymer and solvent.[17] The higher the formation of hydrogen bonds is between solvent and monomers, the more difficulty there will be in volatilizing the solvent. Although the solvent type is an essential factor, other ingredients in adhesive systems can influence solvent volatilization and, consequently, the monomer conversion. For these reasons, the different solvent volatilization methods provided statistically different degrees of conversion means for ethanol- and water-based adhesive systems with similar solvents but differing in their chemical components. For SB and EO, it is likely that a greater formation of hydrogen bonds was achieved so that only active air to evaporate the solvent would be sufficient to break them, increasing solvent volatilization and monomer conversion. This might have not occurred for AM, NB, and XP. For these materials, the degree of conversion of the samples whose solvent was volatilized using either active air for 60 seconds or no air application for 60 seconds was similar. Thus, even in the absence of active air, an extended passive method was probably enough to break the hydrogen bonds, increasing the solvent volatilization and degree of conversion means. This assumption also might be attributed to the water-based adhesive systems tested. While OC obtained the highest monomer conversion after only 60 seconds of air application, OUP already had the highest monomer conversion means after the extended passive method to volatilize the solvent. Although further chemical analyses are necessary to confirm the aforementioned assumptions, the reported results justify the need to evaluate several commercially available adhesive systems in different conditions of solvent volatilization.

Both the acetone-based adhesive systems tested in this study (GO and Stae) presented similar composition, although the components were in different proportions. In addition to the high vapor pressure of acetone, its solubility for hydrogen bonding forces is 7 $(\text{J/cm}^3)^{1/2}$, compared with ethanol, for which it is 20 $(\text{J/cm}^3)^{1/2}$.[18] That is, the affinity of the carbonyl group of acetone to the hydrogen bond with itself or water or any functional group on monomers that are capable of hydrogen bond formation is only about one-third that of ethanol. This is why acetone is so volatile.[17] Thus, it is likely that the solvent might have been volatilized during the adhesive photoactivation, even without using a solvent volatilization technique. Moreover, the heat generated from the curing light also might have facilitated acetone volatilization, yielding statistically similar degrees of conversion for the samples tested in this study. On the other hand, it should be taken into account that no residual moisture from the wet bonding technique was mixed with adhesive solutions, which could completely alter the solvent retention and monomer conversion.[13,19] Thus, clinicians should be encouraged to volatilize the solvent even when using an acetone-based adhesive system, in particular by using more extended times than those recommended by the manufacturers.

CONCLUSION

Therefore, the degree of conversion of the adhesive systems tested was material dependent. The ethanol- and water-based adhesive systems tested benefited from extended solvent volatilization time either with or without air application. The acetone-based adhesive systems tested were not influenced by solvent volatilization techniques.

Conflict of Interest Declaration

The authors of this manuscript certify that they have no proprietary, financial or other personal interest of any nature or kind in any product, service and/or company that is presented in this article.

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