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Influence of Irradiance and Exposure Times on the Mechanical and Adhesive Properties of Universal Adhesives with Dentin

AFM Cardenas • FSF Siqueira • A Nuñez • RF Nonato
KGBA Cavalcanti • CJ Soares • A Reis • AD Loguercio

Clinical Relevance

Better bond strength to dentin and degree of conversion were observed when an adhesive was light-cured using a 5 s * 3200 mW/cm^2, which is in agreement with clinician preference for simplification.

SUMMARY

Objectives: This study evaluated the influence of irradiance/exposure time on the Knoop hardness (KHN) and polymer cross-linking density (PCLD), as well as microtensile bond strength (μTBS), nanoleakage (NL), and *in situ* degree of conversion (DC) of universal adhesives.

Methods and Materials: Two universal adhesive

Andres Felipe Millan Cardenas, DDS, Ms, PhD, professor, Postgraduate Program in Dentistry, CEUMA University, São Luis, MA, Brazil

Fabiana Suelen Figuerêdo de Siqueira, DDS, Ms, PhD, professor, Postgraduate Program in Dentistry, CEUMA University, São Luis, MA, Brazil

Alejandra Nuñez, DDS, Ms, PhD student, Department of Restorative Dentistry, Ponta Grossa State University, Ponta Grossa, Paraná, Brazil, and Professor, Department of Restorative Dentistry and Biomaterials, San Francisco de Quito University, Quito, Ecuador

Rammon de Faria Nonato, DDS, Ms student, Postgraduate Program in Dentistry, CEUMA University, São Luis, MA, Brazil

Karoline Guará Brusaca Almeida Cavalcanti, DDS, Ms, PhD student, Postgraduate Program in Dentistry, CEUMA University, São Luis, MA, Brazil

Carlos José Soares, DDS, Ms, PhD, professor, Departament of Postgraduate Program in Dentistry, CEUMA University, São Luis, MA, Brazil, and Dental School, Department of Operative Dentistry and Dental Materials, Federal University of Uberlândia, Uberlândia, MG, Brazil

*Alessandra Reis, DDS, PhD, professor, Department of Restorative Dentistry, Ponta Grossa State University, Ponta Grossa, PR, Brazil

Alessandro D. Loguercio, DDS, Ms, PhD, professor, Department of Restorative Dentistry, Ponta Grossa State University, Ponta Grossa, PR, Brazil

*Corresponding author: Rua Carlos Cavalcanti, 4748, Bloco M, Sala 64A – Uvaranas, Ponta Grossa, Paraná, Brazil, 84030-900; e-mail: reis_ale@hotmail.com

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systems, Clearfil Universal Bond Quick (CUQ) and Scotchbond Universal Adhesive (SBU), were light-cured using various irradiance/exposure times: 1400 mW/cm² for 5 s (1400*5); 1400 mW/cm² for 10 s (1400*10); 3200 mW/cm² for 5 s (3200*5); and 3200 mW/cm² for 10 s (3200*10). Adhesive disks from each group were used to measure PCLD by KHN. One hundred and twenty-eight human molars were randomly assigned to 16 groups according to the following variables: adhesive system vs adhesive strategies vs radiance/exposure times. After restoration, specimens were sectioned into resin-dentin sticks and tested for μTBS, NL, and DC. The data from PCLD (%), KHN, μTBS (MPa), NL (%), and DC (%) data were subjected to ANOVA and Tukey's test (α=0.05).

Results: Significant reductions in KHN, μTBS, and DC (p=0.00001) values and an increase in NL and PCLD (p=0.00001) values were observed for 3200*10 when compared with other groups. Higher KHN, μTBS, and DC (p=0.000001) values were observed for 3200*5 in comparison with the other groups. The 1400*5 (7 J/cm²) and 1400*10 (14 J/cm²) groups showed intermediate values (p=0.000001).

Conclusion: Although similar results in terms of hardness, polymer cross-linking density and nanoleakage were observed when 5 seconds at 3200 mW/cm² and 10 seconds at 1400 mW/cm² groups were compared, the use of higher irradiance (3200 mW/cm²) for only 5 seconds showed better results in terms of bond strength and degree of conversion for both universal adhesives to dentin. The prolonged exposure time (10 seconds) at the higher irradiance (3200 mW/cm²) showed the worst results.

INTRODUCTION

Current bonding strategies are classified according to how adhesive systems interact with the smear layer, and they are divided into the etch-and-rinse (ER) strategy and self-etch (SE) strategy.[1] However, there is a trend among manufacturers to simplify bonding procedures to satisfy the demand for adhesive procedures that are faster, less technique-sensitive, and more user-friendly.[1]

In this sense, several manufacturers launched in the market "Universal" adhesive systems, which provide dentists with the choice of selecting the adhesion strategy—ER, SE, or an alternative "selective enamel etching," which is a combination of ER strategy on enamel and SE strategy on dentin.[2-4] Universal adhesives are single one-bottle, no-mix adhesive systems that perform equally well with any adhesion strategy and bond adequately to the tooth structure as well as to different restorative materials.[3,5]

However, all simplified one-bottle adhesives are very complex blends of hydrophilic and hydrophobic monomers, water, solvents, and photoinitiators.[6] Consequently, the polymerization of simplified one-bottle adhesives may be adversely affected by the remaining solvent[7,8] and water,[9] mainly because the complete evaporation of solvents after application is clinically difficult.[10] Therefore, simplified one-bottle adhesives are associated with a lower polymerization pattern as compared with multi-step adhesives, which usually include a solvent-free adhesive as the final step.[11]

Although little attention has been given to the polymerization of adhesive systems,[12] some studies have shown that it is possible to increase the adhesive performance as well as reduce the permeability by applying a prolonged exposure time during adhesive light-curing of resin-dentin bond sticks.[11,13-17] However, this increases chair time, which does not comply with the clinician's preference for simplification. Recently, high-irradiance, third-generation, polywave LED curing units were introduced, capable of reaching more than 3000 mW/cm². These new light-curing devices were launched as a solution to increase the restoration thickness and potentially allow shorter polymerization times to achieve optimal photocuring of restorative materials.[18]

The manufacturers of these polywave devices based the effective polymerization process on the exposure reciprocity law,[19,20] and suggest using a curing light that emits a high irradiance could reduce the exposure time. Thus, it is possible that these polywave devices achieve sufficient polymerization using a shorter curing time, because of the increased irradiance.[21] However, most dental adhesive manufacturers indicate a single exposure duration regardless of the material type, shade, or clinical distance from the tip, recommending the specific exposure times for their products that often do not match those of the light manufacturers.[12]

To the best of our knowledge, no study has addressed the effect of different radiant exposures on the mechanical and bonding properties of universal adhesives using a high-power polywave device. Thus, this study aimed to evaluate the influence of radiant exposure on the Knoop hardness (KHN) and polymer cross-linking density (PCLD), as well as the dentin bonding properties (microtensile bond strength [μTBS], nanoleakage [NL], and *in situ* degree of conversion [DC]) of universal adhesives. The null hypotheses tested were that irradiance and exposure

time do not affect (1) the Knoop hardness and polymer cross-linking density, as well as (2) μTBS and DC and (3) NL of universal adhesives bonded to dentin.

METHODS AND MATERIALS

Experimental Design and Calibration Procedures

Two commercial universal adhesive systems were used: Clearfil Universal Bond Quick (CUQ, Kuraray Noritake, Tokyo, Japan) and Scotchbond Universal Adhesive (SBU, 3M Oral Care, St Paul, MN, USA). The detailed composition and batch number of the adhesives are described in Table 1. An LED light-curing unit (Valo, Ultradent Products, Salt Lake City, UT, USA) was used at an irradiance of 1400 mW/cm^2 or 3200 mW/cm^2 for 5 or 10 seconds, resulting in different delivered energy levels (7-32 J/cm^2).

These values were determined using a laboratory-grade spectroradiometer (USB 2000, Ocean Optics, Dunedin, FL, USA) previously calibrated using a NIST-traceable light source and connected to a 6" integrating sphere (Labsphere, North Sutton, NH, USA). For this purpose, the light-emitting area tip end was positioned 1 mm away at the entrance of the integrating sphere, so that all light emitted from the unit was captured. The spectral power measurements were obtained using software (SpectraSuite, v2.0.146, Ocean Optics), where the integrated area was between 350 and 550 nm, which also provided the total emitted power value for that wavelength range. Radiant emittance values of each exposure mode (mW/cm^2) were determined as the total measured power value was divided by the light-emitting area of the distal tip end. This value was then multiplied by the light exposure duration to derive the value of radiant exposure applied to each tooth surface for each light output mode (J/cm^2).

Sample Size

The main outcome of the present study was dentin bond strength. The mean bond strength values of universal adhesives applied to the dentin were considered in the sample size calculation.[4] According to previous literature, the mean bond strength (± standard deviation) of an evaluated universal adhesive was 37 ± 4.0 MPa. To detect a difference of 6 MPa among the tested groups, using $\alpha = 0.05$, a power of 80%, and a two-sided test, the estimated minimum sample size was 8 teeth in each group. The same number of teeth was used for all bonding properties evaluated.

Table 1: *Adhesive Systems, Batch Number, Composition, Groups, and Application Mode*

Adhesive Systems/Batch Number	Composition	Groups	Application Mode	
			Etch-and-rinse	Self-etch
Clearfil Universal Bond Quick (CUQ) Kuraray/ CD0012	10-MDP, BisGMA, HEMA, hydrophilic amide resin monomers, colloidal silica, silane coupling agent, NaF, camphorquinone, ethanol, water	1400 mW/cm^2 for 5 s (1400*5 = 7 J/cm^2) 1400 mW/cm^2 for 10 s (1400*10 = 14 J/cm^2)	1. Apply etchant for 15 s. 2. Rinse thoroughly. 3. Blot excess water; 4. Apply adhesive as for the self-etch mode	1. Apply adhesive to the entire surface with a microbrush and rubbing. No waiting time is required. 2. Dry by blowing mild air for 5 s until the adhesive did not move. 3. Light cure according to experimental groups
Scotchbond Universal (SBU) 3M Oral Care/638367	10-MDP, dimethacrylate resins, HEMA, methacrylate-modified polyalkenoic acid copolymer, nanofiller, ethanol, water, initiators, silane	3200 mW/cm^2 for 5 s (3200*5 = 16 J/cm^2) 3200 mW/cm^2 for 10 s (3200*10 = 32 J/cm^2)	1. Apply etchant for 15 s. 2. Rinse thoroughly. 3. Blot excess water. 4. Apply adhesive as for the self-etch mode.	1. Applied the adhesive to the entire preparation and left undisturbed for 20 s. 2. Direct a gentle stream of air over the liquid for about 5 s until it no longer moves and the solvent evaporates completely. 3. Light cure according to experimental groups.

Abbreviations: 10-MDP, 10-methacryloyloxydecyl dihydrogen phosphate; bis-GMA, bisphenol glycidyl methacrylate; HEMA, 2-hydroxyethyl methacrylate.

Knoop Hardness Test for Polymer Cross-Linking Density

The adhesive disks for the KHN and PCLD tests were produced based on the combination of the main variables, that is: (1) *Adhesives*: CUQ and SBU; (2) *Irradiance/exposure times*: 1400 mW/cm^2 for 5 seconds (7 J/cm^2), 1400 mW/cm^2 for 10 seconds (14 J/cm^2), 3200 mW/cm^2 for 5 seconds (16 J/cm^2), and 3200 mW/cm^2 for 10 seconds (32 J/cm^2); and (3) *Immersion in ethanol*: measurement was performed *before* and *after* immersion in ethanol.

Five adhesive disks of each material were produced in a brass mold 5.0 mm in diameter and 1.0 mm in height (Odeme Prod Odont, Joaçaba, SC, Brazil).[22] The adhesive was dripped into the mold, until filling it completely. The air bubbles trapped in the adhesives were removed with a microbrush. Then, the solvent was evaporated using an air-water syringe for 40 seconds. A polyester Mylar strip was placed on top of the adhesive, which was light-cured according to each irradiance, time, and adhesive group. For standardizing the light-cured procedures, the Valo unit was fixed in a clamp with the emitting end of the light guide 1 mm away from the top surface. After light-curing, the specimens were stored in a dry environment at 37°C for 24 hours.

For measurement of Knoop hardness, indentations were made in the light-cured top surface with a Knoop indenter (KHN, Shimadzu, Kyoto, Japan) using a 10 g load for 15 seconds. Three indentations were made in each specimen, and these values were averaged for statistical purposes. After the first KHN measurement, specimens were stored in a 100% ethanol solution at 37°C for 24 hours, and then the second KHN measurement was performed. The polymer cross-linking density was estimated by the softening effect of the ethanol, that is, by the decrease in hardness.[22]

Selection and Preparation of Teeth

One hundred and twenty-eight extracted, caries-free human molars were used. The teeth were collected after obtaining the patients' informed consent. The teeth were disinfected in 0.5% chloramine, stored in distilled water, and used within 6 months after extraction. The teeth were sectioned parallel to the occlusal surface using a low-speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA) under water-cooling to expose the mid-coronal dentin. A smear layer was standardized for all specimens by grinding each flat dentin surface with #600-grit silicon carbide paper (SiC) under running water for 60 seconds.

Experimental Design

The teeth were then randomly assigned to 16 groups (n=8 dentin specimens for μTBS, NL, and DC) based on the combination of the main variables, that is: (1) *Adhesive*: CUQ and SBU; (2) *Adhesive strategies*: two-step etch-and-rinse (ER) and one-step self-etch (SE) approaches; and (3) *Irradiance/exposure times*: 1400 mW/cm^2 for 5 seconds (7 J/cm^2), 1400 mW/cm^2 for 10 seconds (14 J/cm^2), 3200 mW/cm^2 for 5 seconds (16 J/cm^2), and 3200 mW/cm^2 for 10 seconds (32 J/cm^2). The composition, application mode, and batch numbers are listed in Table 1.

Restorative Procedures

For the ER strategy, a 37% phosphoric acid gel (Condac, FGM Dental Products, Joinville, SC, Brazil) was applied to the dentin surfaces for 15 seconds, followed by rinsing with water for 30 seconds and air-drying for 5 seconds. For the SE strategy, the dentin was not conditioned. For both strategies, the adhesives were applied according to the manufacturer's instructions, and light-cured according to each irradiance/exposure time. For standardizing the light-cured procedures, the Valo unit was fixed in a clamp with the emitting end of the light guide 1 mm away from the occlusal surface. To achieve the exposure times when the device was in high irradiance mode and considering the Valo was set to 3 seconds, the curing time was set to 5 (3 + 2) and 10 (3 × 3 +1) seconds, with the light spot performed immediately in sequence.

The resin composite buildups (DA4, Opallis, FGM, Joinville, Brazil) were then constructed incrementally (three 1.5-mm increments) and each increment was light-cured for 40 seconds each using the same LED light-curing unit (Valo, Ultradent Products) at 1000 mW/cm^2 (40 J/cm^2). A single operator performed all the bonding procedures.

After storage in distilled water at 37°C for 24 hours, the specimens were sectioned longitudinally in the mesio-distal and buccal-lingual directions across the bonded interface, using the low-speed diamond saw to obtain resin-dentin bonded sticks with a cross-sectional area of approximately 0.8 mm^2, as measured with digital calipers (Digimatic Caliper, Mitutoyo, Tokyo, Japan). The number of resin-dentin bonded sticks showing pretest failure (PTF) during specimen preparation was recorded for each tooth.

The resin-dentin bonded sticks obtained for each tooth were randomly distributed as follows: three and two resin-dentin bonded sticks from each tooth and each experimental condition were evaluated, respectively, for NL and DC within adhesive/hybrid layers; the rest of the specimens were tested for μTBS testing. As the

mean μTBS, NL and DC of all resin-dentin bonded sticks from the same tooth were averaged for statistical analyses, and the sample size was eight teeth per group for each experimental group and for each test evaluated (μTBS, NL, and DC tests).

Microtensile Bond Strength Testing

Resin-dentin bonded sticks were attached to a Geraldeli's jig[23] with cyanoacrylate adhesive and tested under tension (Kratos Dinamometros; Cotia, SP, Brazil) at 0.5 mm/min until failure. The μTBS values (MPa) were calculated by dividing the load at failure by the cross-sectional bonding area. The failure modes of the resin-dentin bonded sticks were classified as either cohesive ([C], failure exclusively within the dentin or the resin composite), or adhesive/mixed ([A/M], failure at the resin-dentin interface, or failure at the resin-dentin interface with partial cohesive failure of the neighboring substrates). This classification was performed under a stereomicroscope (Olympus SZ40; Tokyo, Japan) at 100× magnification. The number of specimens with PTF was low, and because of this, it was not included in the average.

Nanoleakage Evaluation

The resin-dentin bonded sticks were immersed in ammoniacal silver nitrate solution according to the protocol previously described by Tay and others.[24] The resin-dentin bonded sticks were placed in the solution in the dark for 24 hours, rinsed thoroughly in distilled water, and immersed in photodeveloping solution for 8 hours under fluorescent light to reduce silver ions to metallic silver grains within the spaces along the bonded interface. The specimens were polished with wet 600-, 1000-, 1200-, 1500-, 2000-, and 2500-grit SiC paper using a polishing cloth. They were then ultrasonically cleaned, air dried, mounted on stubs, and coated with carbon-gold (MED 010, Balzers Union, Balzers, Liechtenstein). Resin-dentin interfaces were analyzed using a field-emission scanning electron microscope operated in backscattered mode (VEGA 3 TESCAN, Shimadzu, Tokyo, Japan). Photomicrographs of representative surface area were taken at 1000× magnification. Three images of each resin-dentin bonded stick were captured.[25,26] The relative percentage of NL within the adhesive and hybrid layers in each specimen was measured in all images using Image J software (National Institutes of Health, Bethesda, MD, USA).[27]

In Situ Degree of Conversion (*in situ* DC) Within Adhesive/Hybrid Layers

The resin-dentin bonded sticks were wet polished, ultrasonically cleaned and positioned in a micro-Raman microscope (XploRA ONE, HORIBA Scientific, Piscataway, NJ, USA), which was first calibrated to zero and then to coefficient values using a silicon sample. The samples were analyzed using a 532-nm diode laser through a 100× air objective. The Raman signal was acquired with 600 lines/mm on a graft centered between 400 and 2000 cm^{-1}. The employed parameters were 100 mW, spatial resolution 3 μm, spectral resolution 1 cm^{-1}, and accumulation time 30 seconds with 5 co-additions. Spectra were obtained at the adhesive-dentin interface at three random sites per bonded stick, within the hybrid layer in the intertubular-infiltrated dentin. Post-processing of the spectra was performed using Opus Spectroscopy Software version 6.5. The average of the values was used for statistical analysis, and the spectra of the uncured adhesives were considered as references.

The ratio of the double-bond content of monomer to polymer in the adhesive was quantified by calculating the ratio derived from the aliphatic C=C (vinyl) absorption (1638 cm^{-1}) to the aromatic C=C absorption (1608 cm^{-1}) signals for both polymerized and unpolymerized samples (n=5). The DC was calculated according to the following formula:

$$\textit{In situ}\ \text{DC}\ (\%) = (1-[R_{\text{cured}}/R_{\text{uncured}}]) \times 100,$$

where "*R*" is the ratio of aliphatic and aromatic peak intensities at 1639 cm^{-1} and 1609 cm^{-1} in cured and uncured adhesives, respectively.[26] In addition, the more intense peaks observed and the corresponding chemical bonds were recorded for all materials.

Statistical Analysis

The mean values of the KHN test were subjected to a three-way ANOVA (adhesive vs irradiance/exposure times vs immersion in ethanol). In addition, the PCLD (%) data were evaluated by two-way ANOVA (adhesive vs irradiance/exposure times).

The mean μTBS (MPa), NL (%), and DC (%) of all bonded sticks from the same tooth were averaged for statistical analyses, ensuring that the experimental unit in the study was the tooth. The number of specimens with PTF was low (ranging between 1.5 to 3%), and because of this, it was not included in the average. The μTBS, NL, and DC mean for each group were obtained from the average of the eight teeth used per group. For the bonding properties, data were subjected to three-way ANOVA (adhesive strategy vs adhesive vs irradiance/exposure times). Finally, for all tests, Tukey's test with a level of significance of 5% was applied.

Table 2: *Knoop Hardness Values (KHN ± Standard Deviation) as an Estimation of Polymer Cross-Linking Density (Δ %) After Immersion in Absolute Ethanol for All Experimental Groups*[a]

Groups	Clearfil Universal Bond Quick						Scotchbond Universal					
	5 s			10 s			5 s			10 s		
	Before	After	Δ (%)	Before	After	Δ (%)	Before	After	Δ (%)	Before	After	Δ (%)
1400 mW/cm²	10.2 ± 0.5 b	5.4 ± 1.5 c	46 B	13.6 ± 1.2 a	8.8 ± 1.4 b	35.4 A	10.1 ± 1.7 b	6.2 ± 1.1 c	44 B	14.2 ± 1.4 a	9.3 ± 0.8 b	35 A
3200 mW/cm²	13.3 ± 1.1 a	8.9 ± 1.2 b	33 A	8.2 ± 1.3 b	2.0 ± 0.6 d	75.1 C	13.7 ± 0.8 a	8.9 ± 1.4 b	35 A	6.4 ± 1.7 c	1.7 ± 0.4 d	73 C

[a] *Different lowercase letters indicate statistically different means in KHN (three-way repeated measured ANOVA and Tukey's test; p<0.005); different uppercase letters indicate statistically different means in estimation of polymer cross-linking density (two-way ANOVA and Tukey's test; p<0.005).*

RESULTS

Knoop Hardness Test for Polymer Cross-Linking Density

The KHN and the estimation of the PCLD are shown in Table 2. Regarding the Knoop values, the cross-product interaction was not significant, as was the main factor adhesive (Table 3). However, the primary factors of irradiance/exposure times and immersion in ethanol were considered statistically significant (Table 3; p=0.000001 and p=0.000001, respectively). Higher KHN values were observed for the 3200*5 (16 J/cm²) and 1400*10 (14 J/cm²) groups, while lower KHN values were observed for 3200*10 (32 J/cm²) when compared to the other groups (Table 2). The 1400*5 (7 J/cm²) group showed intermediate KHN values (Table 2). In contrast, for all groups, a significant decrease in KHN was observed after ethanol immersion (Table 2).

However, a significant decrease in PCLD was observed after ethanol immersion in all groups (Table 2; p=0.00001), with a higher percentage of reduction observed in the 3200*10 (32 J/cm²) group. In contrast, a lower percentage of reduction in the PCLD was measured for the 3200*5 (16 J/cm²) and 1400*10 (14 J/cm²) groups. The 1400*5 (7 J/cm²) group showed intermediate PCLD reduction values (Table 2; p=0.000001).

Microtensile Bond Strength (μTBS)

The majority of the fracture pattern was adhesive and mixed failures (data not shown). Regarding the μTBS values, the cross-product interaction was not significant, as well as the main factor adhesive strategy (Table 4). However, the primary factors, that is, adhesive and irradiance/exposure time were considered statistically significant (Table 4; p=0.000001 and p=0.000001, respectively).

Higher μTBS values were observed for 3200*5, while lower μTBS values were observed for 3200*10 (32 J/cm²) when compared to the other groups (Table 5). Regarding the other groups, for CUQ, there was no significant difference between 1400*5 (7 J/cm²) and 1400*10 (14 J/cm²). In contrast, for SBU, 1400*5 (7 J/cm²) showed lower μTBS values when compared to 1400*10 (14 J/cm²) (Table 5). Overall, SBU showed higher μTBS values when compared to CUQ (Table 5).

Table 3: *Three-Way ANOVA Results (Knoop Hardness Test Values)*

Source of Variation (*)	SS	DF	MS	F	P
Intercept	6322.035	1	6322.035	2267.423	0.000000
Adhesive	0.425	1	0.425	0.152	0.697569
Strategy	502.453	1	502.453	180.207	0.000001
Irrad/time	608.338	3	202.779	72.728	0.000001
Adhesive*Strategy	0.275	1	0.275	0.099	0.754523
Adhesive*Irrad/Time	9.840	3	3.280	1.176	0.325738
Strategy*Irrad/Time	2.340	3	0.780	0.280	0.839815
Adhesive*Strategy*Irrad/Time	2.729	3	0.910	0.326	0.806373
Error	178.445	64	2.788		

Abbreviations: SS, sum of squares; DF, degrees of freedom; MS, mean squares; F, f obtained; p, probability.

Table 4: *Three-Way ANOVA Results (Microtensile Bond Strength Test Values)*

Source of Variation (*)	SS	DF	MS	F	P
Intercept	193565.2	1	193565.2	11190.70	0.000000
Adhesive	1350.5	1	1350.5	78.08	0.000001
Strategy	199.3	1	199.3	11.53	0.095060
Irrad/time	9200.9	3	3067.0	177.31	0.000001
Adhesive*Strategy	0.1	1	0.1	0.01	0.938365
Adhesive*Irrad/Time	115.5	3	38.5	2.23	0.088998
Strategy*Irrad/Time	82.0	3	27.3	1.58	0.198122
Adhesive*Strategy*Irrad/Time	15.1	3	5.0	0.29	0.831774
Error	1937.3	112	17.3		

Abbreviations: SS, sum of squares; DF, degrees of freedom; MS, mean squares; F, f obtained; p, probability.

Nanoleakage Analysis (NL)

The cross-product interaction as well as the main factors of adhesive strategy and adhesive were not significant (Table 6). However, the primary factor of irradiance/exposure time was considered statistically significant (Table 6; p=0.000007). A significantly higher NL value was observed for the 3200*10 group than in the other groups (Table 7 and Figure 1). In addition, a closer view regarding the NL of the ER strategy values showed that significant and lower NL values were observed when 1400*10 (14 J/cm^2) was compared to 1400*5 (7 J/cm^2) (Table 7 and Figure 1). Figure 1 showed the representative photomicrographs obtained for all experimental groups.

Table 5: *Means of Microtensile Bond Strength Values (MPa ± Standard Deviation) for All Experimental Groups*[a]

Groups	Clearfil Universal Bond Quick				Scotchbond Universal			
	Etch-&-rinse		Self-etch		Etch-&-rinse		Self-etch	
	5 s	10 s	5 s	10 s	5 s	10 s	5 s	10 s
1400 mW/cm^2	33.62 ± 4.6 cd	36.87 ± 3.9 c	31.82 ± 4.8 cd	35.15 ± 3.9 c	36.44 ± 3.7 c	46.58 ± 4.5 b	35.39 ± 4.1 c	45.23 ± 4.0 b
3200 mW/cm^2	47.27 ± 4.5 b	25.83 ± 3.9 e	47.03 ± 3.9 b	23.24 ± 4.4 e	54.79 ± 4.5 a	31.80 ± 4.5 cd	53.38 ± 3.3 a	27.39 ± 2.6 de

[a]*Different letters indicate statistically different means (three-way ANOVA and Tukey's test;* p<*0.005).*

Table 6: *Three-Way ANOVA Results (Nanoleakage Test Values)*

Source of Variation (*)	SS	DF	MS	F	P
Intercept	459060.0	1	459060.0	27619.80	0.000000
Adhesive	0.0	1	0.0	0.00	0.988980
Immersion in ethanol	23.7	1	23.7	1.43	0.234974
Irrad/time	496.4	3	165.5	9.96	0.000007
Adhesive*immersion in ethanol	0.5	1	0.5	0.03	0.865061
Adhesive*Irrad/Time	56.1	3	18.7	1.13	0.342109
Immersion in ethanol*Irrad/Time	36.4	3	12.1	0.73	0.536657
Adhesive*immersion in ethanol*Irrad/Time	83.9	3	28.0	1.68	0.174837
Error	1861.5	112	16.6		

Abbreviations: SS, sum of squares; DF, degrees of freedom; MS, mean squares; F, f obtained; p, probability.

Table 7: *Means of Silver Nitrate Deposition (% ± Standard Deviation) for All Experimental Groups*[a]

Groups	Clearfil Universal Bond Quick				Scotchbond Universal			
	Etch-&-Rinse		Self-etch		Etch-&-Rinse		Self-etch	
	5 s	10 s	5 s	10 s	5 s	10 s	5 s	10 s
1400 mW/cm²	11.20 ± 1.9 b	8.23 ± 1.6 a	10.43 ± 1.9 ab	7.35 ± 1.9 a	11.09 ± 1.8 b	6.31 ± 1.7 a	9.37 ± 1.7 ab	5.26 ± 1.1 a
3200 mW/cm²	9.54 ± 1.8 ab	16.38 ± 1.6 c	7.27 ± 1.6 a	15.73 ± 1.5 c	8.39 ± 1.6 ab	15.33 ± 1.7 c	6.11 ± 1.8 a	13.29 ± 1.9 c

[a]*Different letters indicate statistically different means (three-way ANOVA and Tukey's test;* p*<0.005)*

In Situ Degree of Conversion (*in situ* DC) Within Adhesive/Hybrid Layers

Regarding the DC values, the cross-product interaction was not significant, in addition to the main factor adhesive strategy (Table 8). However, the primary factors, that is, adhesive and irradiance/exposure time were considered statistically significant (Table 8; p=0.00002 and p=0.00001, respectively).

Higher DC values were observed for 3200*5, while lower DC values were observed for 3200*10 (32 J/cm²) when compared to the other groups (Table 9). When 1400*5 (7 J/cm²) and 1400*10 (14 J/cm²) were compared, no significant difference was observed for DC. However, overall, SBU showed a majority of higher DC values when compared to those of CUQ (Table 9).

DISCUSSION

Adhesive systems are considered resin-based materials and require light-curing to achieve maximum mechanical properties.[12] However, only a few studies have been carried out to evaluate the effect of polymerization on improving the adhesive properties of dentin.[11,13-17] It seems that, due to the non-opaque yellowish color aspects of the adhesive systems arising from the low amount of filler added,[28] dentists and manufacturers tend to neglect the polymerization time or the irradiance necessary to achieve the correct adhesive polymerization when applied to the cavity.[12] Some studies have reported that the exposure time recommended for dental adhesives is not adequate to obtain an optimal polymerization, even under *in vitro* conditions.[11,13-17]

The polymerization reaction of adhesive systems requires a certain amount of quantum energy to activate the photoinitiator so that it can react with a co-initiator to produce free radicals.[29,30] Therefore, the increase in the amount of quantum energy, through higher irradiance or by increasing the exposure times,

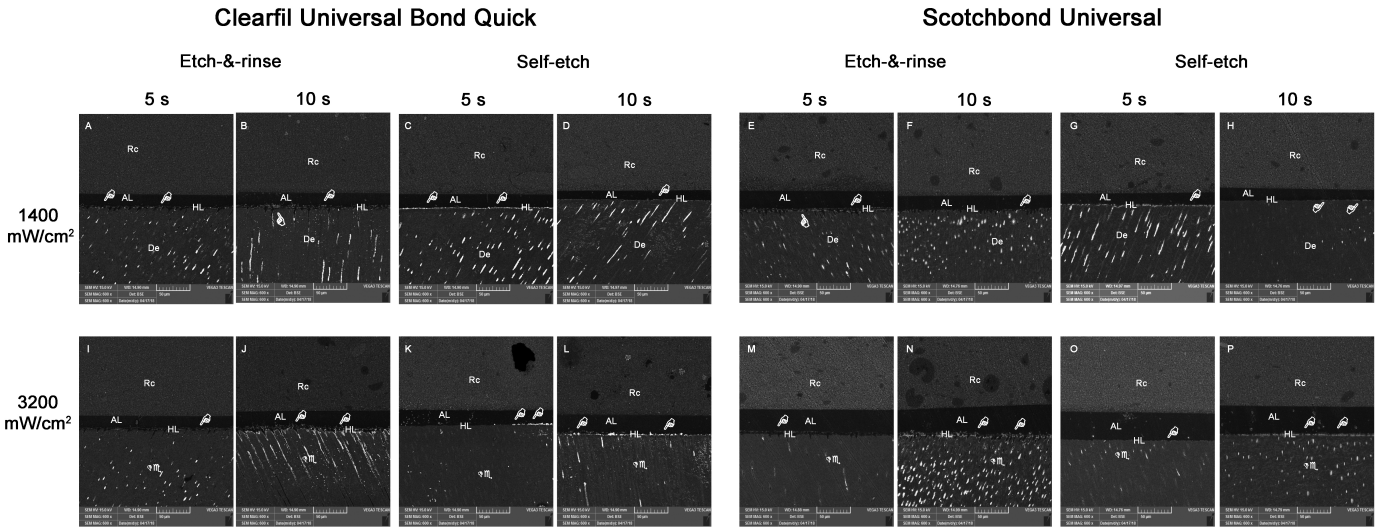

Figure 1. *Photomicrographs obtained by backscattered SEM mode of all experimental groups. Independent of the adhesive system used, it is possible to observe a mild silver deposit at the resin-dentin interface for 1400*10 and 3200*5 groups (hand indicator). For both adhesive strategies and adhesive systems used, the 3200*10 groups demonstrated a more pronounced silver nitrate infiltration (hand indicator). Rc, resin composite; AL, adhesive layer; HL, hybrid layer; De, dentin (micron bar = 50 mm; original magnification = 1000×).*

Table 8: *Three-Way ANOVA Results (*In Situ *Degree of Conversion Within Adhesive/Hybrid Layers Test Values)*

Source of Variation (*)	SS	DF	MS	F	P
Intercept	14746.03	1	14746.03	5347.859	0.000000
Adhesive	69.65	1	69.65	25.259	0.000002
Strategy	0.00	1	0.00	0.000	1.000000
Irrad/time	1333.43	3	444.48	161.196	0.000001
Adhesive*Strategy	0.00	1	0.00	0.000	1.000000
Adhesive*Irrad/Time	0.00	3	0.00	0.000	1.000000
Strategy*Irrad/Time	3.85	3	1.28	0.465	0.707327
Adhesive*Strategy*Irrad/Time	0.00	3	0.00	0.000	1.000000
Error	308.83	112	2.76		

Abbreviations: SS, sum of squares; DF, degrees of freedom; MS, mean squares; F, f obtained; p, probability.

leads to the formation of more free radicals, which initiates polymerization.[29] This may provide room for the formation of a high-molecular-weight cross-linked polymer.[8,13] This better polymerization leads to a less permeable[8,11,14] and more resistant hybrid layer to water degradation.[13,16]

It was observed that an increase in irradiance (3200*5) or exposure time (1400*10) that results in very similar delivered energy (16 and 14 J/cm^2), when compared to the 1400*5 group, significantly improved the DC inside the hybrid layer along with the immediate microhardness (as an indirect measurement of the DC), in agreement with previous studies published with an older generation of adhesive systems.[11,13-17] This could be attributed to a higher amount of quantum energy delivered when an increase of irradiance (3200*5) or exposure time (1400*10) was applied. The greater the amount of quantum energy, the better the polymer formed.[12,30]

Despite all groups experiencing a considerably decreased percentage of PCLD after immersion in ethanol, a significantly higher percentage of PCLD was observed for the 1400*5 (7 J/cm^2) group when compared to the 3200*5 (16 J/cm^2) or 1400*10 (14 J/cm^2) groups. The PCLD is considered an indirect test to measure a polymer network by softening.[22,31,32] This is accepted as an appropriate test, because highly cross-linked polymers are more resistant to degradation and solvent uptake, whereas linear polymers present more space and pathways for solvent molecules to diffuse within their structure.[33] When an inadequate curing time is applied, there is a risk of lower cohesion in the network due to less cross-linking and secondary forces.[12] Therefore, the results obtained in the 1400*5 (7 J/cm^2) group could be attributed to a lower cross-linking density, due to the insufficient energy delivered.

Another factor that cannot be ruled out is the increase in the temperature produced by the high-energy polywave light-curing units,[18,34-36] maximized by an increase in irradiance (3200*5 [16 J/cm^2]) or by prolonged exposure time (1400*10 [14 J/cm^2]). Mouhat and others[34] measured the superficial temperature of different radiant exposures. At 7 J/cm^2, a radiant exposure similar to that obtained for the 1400*5 group, the temperature values measured were around 39.6°C. Otherwise, an increase of 3.2 to 4.1°C was observed when radiant exposure ranged between 14 and 15 J/cm^2. These last radiant exposures are similar to those produced by the 3200*5 (16 J/cm^2) and 1400*10 (14 J/cm^2) groups.

Table 9: *Means of* In Situ *Degree of Conversion Values (% ± Standard Deviation) for All Experimental Groups*[a]

Groups	Clearfil Universal Bond Quick				Scotchbond Universal			
	Etch-&-rinse		Self-etch		Etch-&-rinse		Self-etch	
	5 s	10 s	5 s	10 s	5 s	10 s	5 s	10 s
1400 mW/cm^2	56.49 ± 1.0 cd	60.34 ± 1.9 c	55.44 ± 1.8 cd	59.89 ± 1.2 c	64.21 ± 1.8 b	65.14 ± 1.4 b	58.33 ± 1.7 c	59.22 ± 2.2 c
3200 mW/cm^2	67.25 ± 1.0 b	51.29 ± 1.3 d	65.38 ± 1.4 b	50.31 ± 1.7 d	71,36 ± 1.0 a	45.32 ± 1.3 e	69.60 ± 1.3 a	45.56 ± 1.1 e

[a] *Different letters indicate statistically different means (three-way ANOVA and Tukey's test;* p<*0.005).*

In addition, the increase in temperature can be responsible for favoring the evaporation of solvents from the material,[13] as is known from the benefits of heat in the evaporation of solvents, mainly when using warm-air drying for solvent evaporation.[37,38] Reis and others,[13] based on thermogravimetric analysis, hypothesized that the extra heat and energy produced by the increase in the light exposure could have favored the evaporation of solvent and water, but also increased the degree of conversion of the material,[8,13,14,16] thereby reducing the amount of residual low molecular weight monomers and oligomers.

All of these together might be considered sufficient reasons for the higher resin–dentin bond strengths observed in the present investigation, when an increase in irradiance (3200*5 [16 J/cm^2]) or prolonged exposure time (1400*10 [14 J/cm^2]) was applied in comparison to the 1400*5 (7 J/cm^2) group for both universal adhesives and adhesive strategies, leading the authors to reject all null hypotheses.

Despite a very close radiant exposure for the 3200*5 (16 J/cm^2) and 1400*10 (14 J/cm^2) groups, the former showed a higher DC value inside the hybrid layer and a higher μTBS when compared to the latter. For both adhesives and adhesive strategies, the irradiance was more important for improving the adhesive properties than the increase in polymerization time. In other words, for both adhesive systems used, it seems that the higher irradiance led to an optimum rate of initiation that produced the highest quantum yield and, consequently, better mechanical properties. On the other hand, this may not be enough to form a highly cross-linked polymer, as both groups showed a similar percentage of PCLD. Future studies need to be conducted to confirm the present hypothesis.

However, the most unexpected result was obtained for the 3200*10 group. All properties evaluated were negatively influenced by the higher radiant exposure used (32 J/cm^2). It is known that during the photopolymerization of monomers, a significant increase in temperature occurs in a short exposure time, even in thin films such as adhesive systems, especially when ultrafast polymerization is used.[39] As a result, the molecular mobility increases, particularly in the gelation stage, thus lifting some of the migration restrictions of the reactive species, which are known to be primarily responsible for the premature ending of the polymerization.[39]

Independent of the radiant exposure, among high-power light-curing units, the Valo device produces the greatest increase of temperature[18,34-36] which seemed to directly affect the results of the 3200*10 group. The use of radiant exposure at approximately 32 J/cm^2 significantly increased the superficial temperature to 48.2°C, while an increase of 5°C was seen for 14-16 J/cm^2 and 8.6°C for 7 J/cm^2 when compared with the superficial temperatures evaluated in the other groups of this study.[34] Thus, we speculate that this increase in temperature, due to a large amount of heat received in the 3200*10 group, substantially impaired the adhesive system polymerization, leading to low KHN values and a greater reduction in the percentage of PCLD as well as low μTBS and higher NL values, leading to further rejection of all null hypotheses.

Moreover, the increased irradiance of the 3200*10 group not only produced adhesive interfaces with a high number of potential failures, but the elevated temperature during polymerization may also result in the generation of heat within the tooth, pulp chamber, and surrounding tissues.[18,34-36] For example, a higher temperature has been observed in the pulp chamber when an adhesive was light-cured in comparison with light-curing of composite or base/liner materials.[35,40] Therefore, when an adhesive is applied as the first layer in medium or deep cavities, during the light-curing procedure, the tooth should be air-cooled during the photocuring procedure, or an interval of 1-2 seconds should be included after every 5-10 seconds of light exposure, as indicated by Strassler and Price.[41] It is worth mentioning that the manufacturer of Valo did not recommend 10 seconds for light-curing any resin-based material when applied in a "plasma/turbo" mode.

In this study, two universal adhesive systems were tested, with SBU demonstrated to have higher values in terms of bond strength and DC values when compared to CUQ. The former is the only universal adhesive to be previously evaluated, which contains two functional monomers: 10-MDP (10-methacryloyloxydecyl dihydrogen phosphate) and methacrylate-modified polyalkenoic acid copolymer[42,43] which potentiate the chemical interaction of the SBU with the tooth structure.[44] In addition, the application procedures between both materials are different. CUQ is used without waiting for the adhesive to interact with the bonding substrate (the "no-waiting" concept).[45] This can influence the DC results, due to the low time for solvent evaporation, as was observed in the present study, even with increasing radiant exposure (7 to 14-16 mW/cm^2). However, the increase in the DC only affected the bond strength results, since no significant difference was observed in the NL or PCLD results when both adhesives were evaluated.

Finally, it is worth mentioning that, according to a review published by Cadenaro and others,[12] the adequate polymerization of an adhesive has been clearly

correlated with its stability. Therefore, future studies need to be conducted to evaluate the effectiveness of increasing the radiant exposure on the long-term bonding performance of universal bonding adhesives to dentin.

CONCLUSIONS

Similar results in terms of KHN, PCLD and NL were observed when 5 seconds at 3200 mW^2 and 10 seconds at 1400 mW/cm^2 groups were compared. The use of higher irradiance (3200 mW/cm^2) for only 5 seconds showed better results in terms of bond strength and degree of conversion for both universal adhesives to dentin. The prolonged exposure time (10 seconds) at the higher irradiance (3200 mW/cm^2) showed the worst results.

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Regulatory Statement

This study was conducted in accordance with all the provisions of the human subjects oversight committee guidelines and policies of State University of Ponta Grossa under protocol #3.542.383.

Conflict of Interest

The authors of this article 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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