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Original Article

How do increased irradiance and exposure time affect the repair bond strength of universal adhesives to CAD-CAM composite material?

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Abstract

Purpose: This study was designed to investigate the effects of increased exposure time and different levels of irradiance on the repair bond strength (μTBS) and degree of conversion (DoC) of two universal adhesives.

Methods: Computer-aided design-computeraided manufacturing (CAD-CAM) composite blocks were prepared and subjected to thermal cycling. They were randomly divided into two groups based on the adhesive agent used: Clearfil S^3 Bond Universal (CSU) or Gluma Bond Universal (GBU). Within each group, the specimens were further classified into six subgroups (n = 6 blocks) according to irradiance (1,000 or 1,400 mW/cm²) and exposure time (10, 15, or 20 s). The bonded specimens were then repaired using nano-hybrid resin composite. Sticks measuring 0.9 mm² were obtained from the repaired blocks and subsequently tested in a micro-tensile testing machine. Additionally, the DoC was assessed using micro-Raman spectroscopy.

Results: The 1400*10 group of GBU and CSU demonstrated higher μTBS than the 1000*10 and 1400*15 groups, regardless of thermal cycling (P = 0.000). For CSU, the 1400*10 group showed the highest DoC ($P < 0.05$). However, the DoC of GBU did not differ significantly among the various irradiation/exposure times ($P > 0.05$).

Conclusion: The polymerization of adhesives at an irradiance level of 1,400 mW/cm^2 for 10 s (14 J/cm^2) increases the repair μTBS and DoC regardless of thermal cycling.

Keywords: CAD-CAM block, curing light, polymerization, repair, universal adhesive

Introduction

Direct and indirect resin composites are extensively utilized in modern dentistry for the restoration of posterior teeth [1]. Although direct resin composite restorations allow the use of additive restorative techniques and minimally invasive procedures [2], these advantages do not eliminate certain disadvantages, such as polymerization shrinkage stress and relatively low wear resistance [3]. Polymerization shrinkage stress is lower for indirect than for direct composite restorations [1]. In addition, proximal contact, anatomical form and esthetics can be achieved more successfully with indirect composite restorations [1]. Although indirect resin composites can be fabricated and designed in dental laboratories or in the dental office [2], recent interest in the use of computer-aided design-computeraided manufacturing (CAD-CAM) technology for constructing indirect restorations has increased significantly [3]. Since the composite blocks used in CAD-CAM technology are industrially polymerized under high pressure and temperature, they have a higher degree of conversion (DoC), less discoloration, and greater wear resistance than conventionally polymerized direct resin composites [2,4-8]. They also offer several advantages over ceramic materials: they are easier to repair intraorally [9], cause less wear to opposing teeth [4,10], and do not require firing or complex laboratory procedures [4,6,7]. Moreover, they demonstrate comparable esthetic and mechanical performance [11]. Nevertheless, internal stresses caused by factors such as diet, temperature, and pH change, as well as parafunctional habits, can lead to fractures in restorations regardless of the restorative material employed [12]. A recent meta-analysis has reported that indirect restorations exhibit higher annual failure rates (0%-15.5%) than direct restorations (0%-5.4%) [13]. Moreover, chipping and fracture have been identified as the most common causes of failure [13]. These findings highlight the need for an evidence-based repair protocol for management of such clinical conditions.

Universal adhesives, also called multi-mode adhesives, can bond to dental hard tissues with different adhesion strategies (e.g. self-etch, etch-and-rinse or selective enamel etch) according to each specific clinical situation [5,7]. Some universal adhesives can bond to different substrates such as resin composite, glass ceramic and metal without the need for an extra primer or silane agent [6]. This provides a simplified approach for repair of fractures in restorations [8]. Universal adhesives are a complex combination of hydrophilic and hydrophobic monomers, water, solvents and photoinitiators [4]. Water and solvents contained in universal adhesives are responsible for ionizing hydrophilic acidic monomers such as MDP [10]. The air drying times recommended by manufacturers may not allow the adhesive to completely evaporate solvents after application [12]. Because of this, residual solvent and water can also adversely affect the polymerization of universal adhesives [14]. Therefore, one clinical alternative may be to increase the polymerization time to reduce hydrolytic degradation at the adhesive interface [15,16].

Breschi et al. [17] and Cadenaro et al. [15] have reported that the DoC of adhesives increases whereas the permeability of the adhesive layer decreases when adhesives are polymerized for longer than the time recommended by the manufacturers. Clinicians may find prolonged adhesive polymerization more user-friendly since it does not introduce an extra step in the restorative treatment process. Some previous studies have indicated that increasing the level of irradiance (3,200 mW/cm^2-5 s, 16 J/cm^2) and exposure time (1,400 mW/cm^2-10 s, 14 J/cm^2) during the polymerization of universal adhesives increases the dentin bond strength and DoC, while the permeability of the hybrid layer decreases [18,19]. However, the impact of universal adhesive polymerization using a prolonged exposure time and increased level of irradiance on the repair bond strength of CAD-CAM composite materials does not appear to have been studied previously.

Against this background, the objective of the present study was to examine the effects of prolonged exposure time and increased irradiance on the repair bond strength (micro-tensile bond strength, μTBS) and in-situ DoC of universal adhesives, both immediately and after TC. The null hypothesis was that an increase in the exposure time and different irradiance levels during polymerization would not influence (1) the μTBS or (2) the DoC of universal adhesives.

Materials and Methods

CAD-CAM blocks (9 × 7 × 6 mm) (Cerasmart 270 A2-LT 14; GC Corp., Tokyo, Japan) were cut with a low-speed water-cooled cutting device (Micracut 152, Metkon, Bursa, Turkey). The bonding surface of all specimens was polished with 600-grit rotational SiC paper (Forcipol 102, Metkon) at 150 cycles per minute under water cooling for 30 s to achieve a standardized surface [20]. All specimens were subjected to thermal cycling (TC; 10,000 cycles, 5ºC-55ºC, 30 s dwell time) [20].

Specimens were randomly divided into twelve groups (n = 6 blocks)

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Table 1 Material components and application procedures

Material/Batch number	Composition	Application procedure
Clearfil S^3 Bond Universal (CSU; Kuraray Noritake Dental Inc., Tokyo, Japan) 4M006D	Bis-GMA (15-35%), 10-MDP, HEMA (10-35%), ethanol (<20%), water, colloidal silica, dl-camphorquinone, hydrophilic aliphatic dimethacrylate, initiators, accelerators, silane coupling agent	apply adhesive (rubbing): 10 s air pressure (mild): 5 s light curing: per experimental group
Gluma Bond Universal (GBU; Heraeus Kulzer, Hanau, Germany) M010061	10-MDP, 4-META, dimethacrylate resins, acetone, fillers, initiators, silane, water	apply adhesive (rubbing): 20 s air pressure: until liquid no longer moves light curing: per experimental group
Clearfil Majesty Esthetic (Kuraray Noritake Dental Inc.) 3F0243	Bis-GMA, hydrophobic aromatic dimethacrylate, dl-camphorquinone, initiators, silanated barium glass (average particle size 0.7 mm) and pre-polymerized organic filler	composite layer thickness: 1.5 mm light curing: 20 s per layer (20 J/cm²)
Cerasmart 270 (A2-LT/14; GC Corp., Tokyo, Japan) 2304241	Bis-MEPP, UDMA, dodecyl dimethacrylate with 71 wt% silica (20 nm) and barium glass nanoparticles (300 nm)	

based on the combination of two main factors: adhesive (2 levels) and increased irradiance/exposure time (6 levels). Two types of universal adhesive were utilized in this study: (1) Clearfil S^3 Bond Universal (CSU; Kuraray Noritake Dental Inc., Tokyo, Japan) and (2) Gluma Bond Universal (GBU; Heraeus Kulzer, Hanau, Germany).

To simulate clinical conditions, the repair surface of the CAD-CAM composite was roughened parallel to the surface using an extra-fine diamond bur (#837 KREF, Komet; Lemgo, Germany) mounted on a high-speed handpiece operating at 200,000 rpm. Subsequently, the surface was rinsed with an air-water spray for 30 seconds and then dried with oil-free air for 10 seconds.

Universal adhesives were applied according to the manufacturer's instructions, as described in Table 1. The light polymerization step was performed at 1,000 mW/cm^2 for 10 s (10 J/cm^2), 15 s (15 J/cm^2), 20 s (20 J/cm^2); 1,400 mW/cm^2 for 10 s (14 J/cm^2), 15 s (21 J/cm^2), 20 s (28 J/cm^2) using a light-emitting diode polymerization device (VALO Cordless, Ultradent, South Jordan, UT, USA). The adhesives were polymerized at different radiant energy levels (10-28 J/cm^2). The light power output was verified using a radiometer (Hilux Ultra Plus Curing Units, Benlioglu Dental, Ankara, Turkey) before adhesive polymerization.

A polyvinyl siloxane mold (9 × 7 × 12 mm) was used to place the resin composite (Clearfil Majesty Esthetic, Kuraray Noritake Dental Inc.) on the repair surface of the CAD-CAM composite. The resin composite was placed in 1.5-mm layers and polymerized with a light-emitting diode (1,000 mW/cm^2) for 20 s (20 J/cm^2). The entire repair procedure was performed by a single operator (F.B.). Thereafter, the polyvinyl siloxane mold was removed and the blocks were stored in distilled water at 37ºC for 24 h.

CAD-CAM resin composite repair blocks were cut both horizontally and vertically from the adhesive interface using a low-speed cutting device with water cooling. Consequently, CAD-CAM resin composite sticks with a cross-sectional area of approximately 0.9 × 0.9 mm² were produced. Sticks with voids or bubbles evident under a stereomicroscope (Stemi 305; Carl Zeiss Microscopy, Göttingen, Germany) were excluded from the study. Peripheral sticks were removed, resulting in approximately 12 to 15 sticks from each CAD-CAM-composite repair block. Additionally, each stick was measured using a digital caliper (SL-1112, Insize, Suzhou, Jiangsu, PR China) to determine the adhesive surface area.

Microtensile bond strength test

Half of the sticks from the CAD-CAM resin composite repair block were subjected to Microtensile bond strength (μTBS) testing after 24 h, while the other half were exposed after TC (10,000 cycles, 5ºC-55ºC, 30 s dwell time). The sticks with CAD-CAM composite on one half and resin composite on the other were fixed to the jig using cyanoacrylate gel (Loctite 454, Henkel, Düsseldorf, Germany), ensuring that the adhesive interface was centered in the jig. Each stick underwent tensile testing in a micro-tension tester (Bisco AS T-61010K, Schaumburg, IL, USA) at a cross-head speed of 1 mm/min until fracture occurred. The load at failure (in Newtons) was converted to megapascals (MPa) by dividing by the adhesive surface area (mm^2). The load for premature failures, if any, was considered to be 0 MPa and included in the data analysis. Failure modes were examined by a single researcher (M.K.) under a stereomicroscope at ×40 magnification and categorized as adhesive (between CAD-CAM composite and resin composite), cohesive (within CAD-CAM composite and resin composite), or mixed (both adhesive and cohesive). One fracture specimen from each group was taken randomly and examined under a scanning electron microscope (SEM; JSM-6610, JEOL, Peabody, MA, USA). The specimen surfaces were sputter-coated with 99% pure gold and then observed using an acceleration voltage of 15 kV in a vacuum environment at ×60 magnification. CAD-CAM composite blocks (n = 3) were also used as a positive control group to evaluate the cohesive strength of the CAD-CAM composite before and after TC.

In situ degree of conversion

Samples were prepared following the μTBS test protocol. The in situ DoC test did not include TC. Three blocks were utilized for each of the 12 experimental groups (Adhesive, 2 levels; increased irradiance/exposure time, 6 levels). Each CAD-CAM composite-resin composite repair block was cut longitudinally along the adhesive interface with a low-speed water-cooled cutting device. The slices obtained from the repair blocks were polished with 1500- and 2000-grit rotational SiC paper respectively and the sticks were then cleaned in an ultrasonic cleaner for 10 min [21].

The micro-Raman spectrometer (Alpha 300R, WITec Instruments Corp., Ulm, Germany) was first calibrated for zero and then for coefficient values using a silicon sample. Samples were examined under the following micro-Raman parameters: 20 mW neon laser, wavelength 532 nm, spatial resolution approximately 3 mm, spectral resolution approximately 5 cm^{-1}, accumulation time 30 s with 6 co-additions, magnification ×100, beam diameter approximately 1 μm. Spectra were obtained from the adhesive interface at random sites. Two spectra were collected from each repair slice. The average of the spectra recorded for each block was used for statistical analysis.

The spectra of uncured adhesives were obtained to serve as a baseline reference. The specialized Opus Spectroscopy Software version 6.5 was employed to post-process the spectra. The conversion rates were determined by calculating the ratios of C for C - C and C = C absorbance strengths (% of unobserved double bonds) before and after photopolymerization according to the following formula:

$$DoC\ (\%) = \left(1 - \frac{C_{aliphatic} / C_{aromatic}}{U_{aliphatic} / U_{aromatic}}\right) \times 100$$

where the 1,638 cm^{-1} absorption peak of the cured resin is represented by $C_{aliphatic}$, the 1,607 cm^{-1} absorption peak of the cured resin is represented by $C_{aromatic}$, the 1,638 cm^{-1} absorption peak of the uncured resin is represented by $U_{aliphatic}$, and the 1,607 cm^{-1} absorption peak of the uncured resin is represented by $U_{aromatic}$.

Statistical analysis

The number of specimens per group (n = 6) for analysis of repair bond strength was determined on the basis of previous studies [8]. Subsequently, the sample size was verified using F tests in the G*Power software package (version 3.1; Heinrich Heine University, Germany). The sample size was determined to be adequate for this study (power (1-β) = 0.90, effect size = 0.50).

The normality and homogeneity of variances were verified using the Shapiro-Wilk and Levene tests, respectively (P > 0.05). Three-way ANOVA (adhesive, irradiance/exposure time, and TC) and a post hoc Tukey test were utilized to evaluate the μTBS values. Two-way ANOVA (adhesive and irradiance/exposure time) and a post hoc Tukey test were employed to assess the in situ DoC. Statistical analyses were conducted using the SPSS software package (v29.0; IBM, Chicago, IL, USA), with the level of statistical significance set at 0.05.

Table 2 Mean microtensile bond strengths (MPa ± standard deviation) in all experimental groups after 24 h and 10,000 cycles of thermal cycling

		1,000 mW/cm²			1,400 mW/cm²		
		10 s	15 s	20 s	10 s	15 s	20 s
CSU	24 h	33.56 ± 16.63^{Ca}	37.99 ± 10.88^{Aa}	38.63 ± 8.03^{Aa}	39.02 ± 11.37^{Aa}	36.52 ± 9.51^{ABCa}	34.74 ± 10.35^{BCa}
	10,000 TC	33.5 ± 16.11^{ABa}	33.44 ± 19.53^{ABb}	34.66 ± 8.05^{ABa}	36.27 ± 10.02^{Aa}	30.69 ± 9.97^{Bb}	34.52 ± 7.53^{ABa}
GBU	24 h	29.26 ± 10.78^{BCab}	29.70 ± 9.87^{BCc}	29.51 ± 11.15^{BCb}	33.88 ± 8.52^{Ab}	28.00 ± 10.90^{Cb}	32.61 ± 8.37^{ABa}
	10,000 TC	28.55 ± 8.41^{ABb}	29.41 ± 7.64^{ABc}	29.28 ± 9.36^{ABb}	31.41 ± 8.58^{Ab}	27.27 ± 9.19^{Bb}	30.69 ± 10.39^{ABa}

Different uppercase letters in the horizontal lines indicate a significant difference. Different lowercase letters in the vertical lines indicate a significant difference ($P < 0.05$).

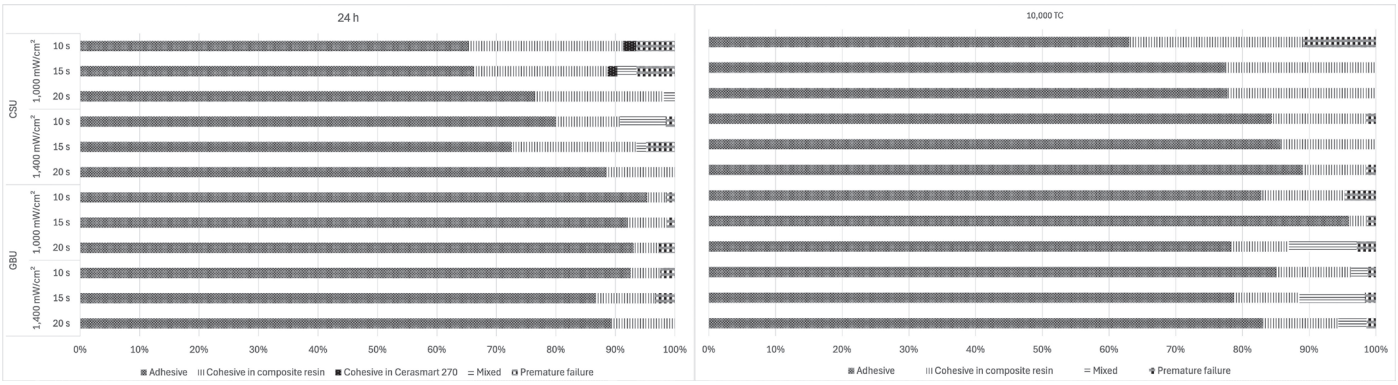


Fig. 1 Stereomicroscope failure analyses of the experimental groups

Results

µTBS analysis

Table 2 presents the means and standard deviations of µTBS (MPa) for the experimental groups. Three-way ANOVA revealed that the adhesive agent ($P = 0.000$, $F = 79.997$), irradiance/exposure time ($P = 0.000$, $F = 4.507$), and TC ($P = 0.002$, $F = 9.815$) significantly affected µTBS. The interactions between the adhesive agent and irradiance/exposure time ($P = 0.092$, $F = 1.895$), adhesive agent and TC ($P = 0.310$, $F = 1.033$), and irradiance/exposure time and TC ($P = 0.277$, $F = 1.264$) were not significant. The triple interaction among the factors was also not significant ($P = 0.085$, $F = 1.939$).

TC significantly decreased µTBS for CSU ($P = 0.005$) but not for GBU ($P = 0.117$), regardless of the irradiance/exposure time. For CSU, the µTBS in the 1000*15 and 1400*15 groups exhibited a significant decrease after TC ($P = 0.006$, $P = 0.004$, respectively).

Overall, CSU demonstrated higher µTBS values than GBU ($P = 0.000$), regardless of irradiance/exposure time and TC. Additionally, the 1400*10 group showed higher µTBS than the 1000*10 ($P = 0.000$) and 1400*15 ($P = 0.000$) groups, regardless of the experimental groups.

The percentages of failure modes at 24 h and 10,000 TC for the experimental groups are shown in Fig. 1. Adhesive failure was the most common failure type at both 24 h and 10,000 TC. Representative SEM images of the fractured surfaces are shown in Fig. 2. The SEM images indicated that most failures identified as adhesive failures when examined with a stereomicroscope were actually mixed failures.

The cohesive strength of the CAD-CAM composite blocks used as a positive control group was 114.34 ± 12.45 MPa at 24 h and 102.17 ± 18.54 MPa at 10,000 TC. In addition, the difference in cohesive strength of the CAD-CAM composite block after TC was statistically significant ($P = 0.020$).

In situ DoC analysis

Table 3 displays the means and standard deviations of the DoC (%) for the experimental groups. The adhesive agent did not significantly affect the DoC ($P = 0.653$, $F = 0.208$). However, the effects of irradiance/exposure time ($P = 0.009$, $F = 1.599$) and the interaction between the adhesive and irradiance/exposure time ($P = 0.013$, $F = 2.336$) were significant.

For CSU, the 1400*10 group demonstrated the highest DoC, and the difference from the other groups was statistically significant, except for the CSU 1000*15 group. The DoC for GBU at different irradiance/exposure times was statistically similar in all experimental groups. In the 1000*15 and 1400*10 groups, CSU exhibited a higher DoC than GBU ($P = 0.034$ and $P = 0.036$, respectively). Additionally, the 1400*10 group showed a significantly higher DoC except for the 1000*15 group, regardless of universal adhesive.

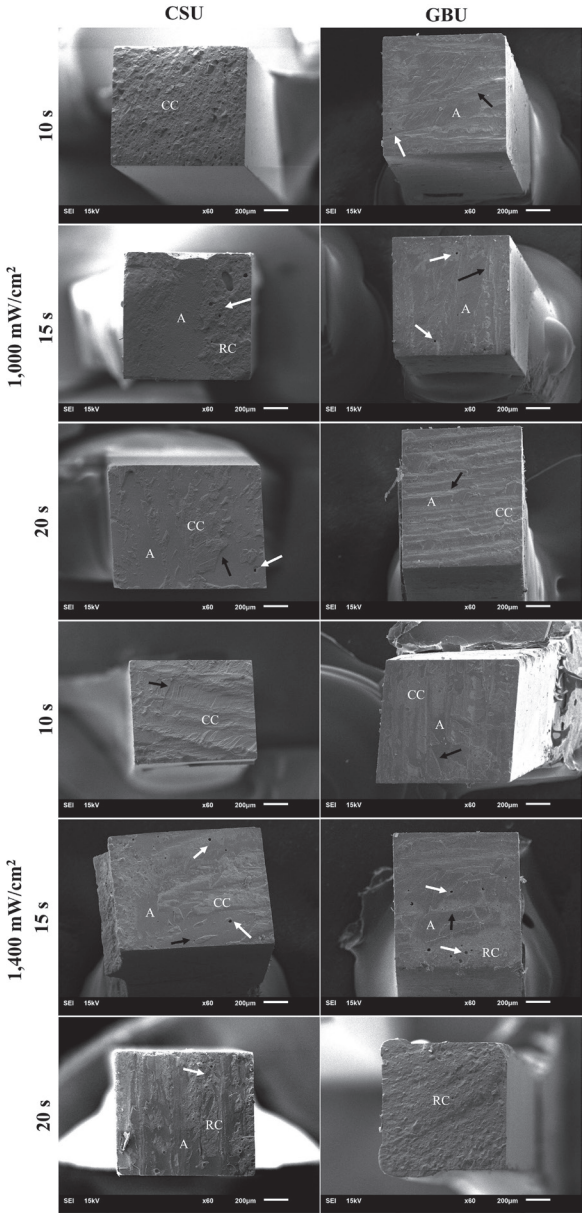


Fig. 2 Representative SEM images of failure modes on the CAD/CAM side after µTBS testing; CC, CAD-CAM resin composite (Cerasmart 270); RC, Resin composite, A, Adhesive layer; White arrows, voids in resin composite; Black arrows, Traces of fractures in the adhesive layer.

Table 3 Means of in situ degree of conversion (% ± standard deviation) for all experimental groups after 24 h

	1,000 mW/cm²			1,400 mW/cm²		
	10 s	15 s	20 s	10 s	15 s	20 s
CSU	72.23 ± 2.72^{Ba}	81.23 ± 2.69^{Aa}	75.81 ± 2.33^{Ba}	81.95 ± 2.41^{Aa}	75.16 ± 3.75^{Ba}	76.63 ± 1.13^{Aa}
GBU	72.27 ± 2.35^{Aa}	76.37 ± 3.54^{Ab}	75.72 ± 2.26^{Aa}	76.84 ± 2.71^{Ab}	75.24 ± 3.39^{Aa}	75.29 ± 1.65^{Aa}

Different uppercase letters in the horizontal lines indicate a significant difference. Different lowercase letters in the vertical lines indicate a significant difference ($P < 0.05$).

Discussion

To optimize the final performance of the adhesive and ensure it exhibits superior mechanical properties, it must be properly polymerized with light [14,22]. However, no previous studies have evaluated the effect of adhesive polymerization on repair bond strength, and those attempting to improve repair bond strength have generally focused on surface preparation procedures. The aim of using universal adhesives in the repair process is to exploit the effects of the silane they contain or to follow common clinical practice. As a result, clinicians might not fully consider the technical precision required for optimal polymerization [14]. Some studies have indicated that the suggested exposure times for dental adhesives are inadequate for achieving optimal polymerization, even under in vitro conditions [15-17]. However, photopolymerization is a more complicated clinical procedure than it appears [14].

Chemical adhesion is essential for creating a durable bond between an old restoration and a new resin composite [23]. Due to the high viscosity of the repair composite, the potential for chemical bonding with the existing restorative material and infiltration into surface irregularities is restricted [24,25]. Therefore, an adhesive agent is essential for creating a strong bond between an old and a new restorative material [26]. Adhesives improve bonding to the resin matrix and exposed filler, allowing micromechanical interlocking by penetrating the micro-irregularities of aged materials, which can result in increased bond strength [26].

The polymerization of adhesives, which are light-curing resin materials, begins by exposing the photoinitiator in their content to photocuring light with adequate irradiance at an appropriate wavelength for a sufficient time [14,15]. Similar to all resin materials, adhesive systems also develop their final properties through a polymerization process characterized by the conversion of carbon-carbon double bonds in monomers to carbon-carbon single bonds, connecting one monomer to another. This process transforms the initial monomers into a durable and robust polymer by creating long chains and cross-links [14].

In this study, the increase in μTBS and DoC values in the 1400*10 (14 J/cm²) group is consistent with previous studies conducted on different adhesives substrates [18,19]. Higher irradiance or increased exposure time results in an increase of quantum yield, leading to formation of more free radicals [27]. This improved polymerization creates an adhesive interface that is less permeable [15,17] and more resistant to water degradation [16]. The greater success observed in the 1400*10 (14 J/cm²) group can be explained by these factors. However, an excessive increase in quantum yield can result in contradictory outcomes. High levels of radiant exposure can cause a significant rise in temperature [28], which can accelerate the initiation rate, leading to an adhesive interface with lower quantum efficiency and, as a result, reduced stability [19].

Interestingly, even though the radiant exposure obtained in the 1000*15 (15 J/cm²) group was nearly identical to that in the 1400*10 (14 J/cm²) group, there was no significant difference in μTBS between the 1000*15 group and the other experimental groups regardless of universal adhesive time and TC. This may indicate that polymerization is much more complex than information available in the literature would suggest. Although the amount of energy delivered to the adherent surface remains the same, variations in irradiance, exposure time, or adherents do not result in similar degrees of success at the adhesive interface. However, it is well accepted that for enhancement of adhesive properties, irradiance is more critical than an increased polymerization time [18]. All of these factors can be considered to account for the more stable adhesive interface observed in the 1400*10 group, allowing rejection of the null hypothesis.

In this study, CSU showed a higher μTBS in comparison to GBU. Differences in the compositions of universal adhesives lead to differences in the properties of adhesive interfaces [29]. Along with the adhesive content, many factors including substrate properties, operator technique, and the characteristics of the light-curing unit, play a role in photopolymerization [14]. CSU used in this study contains ethanol and water solvents, whereas GBU contains acetone/water solvents. Under ideal conditions, it is desirable for the solvents in the adhesive composition to evaporate completely; however, this is very difficult to achieve clinically [30]. The presence of residual solvents at the adhesive interface can negatively affect bond strength, mechanical properties, and DoC [31]. Among common solvents, ethanol and water have a strong tendency to form hydrogen bonds not only with each other but also with resin monomers, which can hinder complete polymerization. Moreover, due to its lower vapor pressure compared to acetone, ethanol evaporates more slowly. This makes the removal of solvents from ethanol-based adhesives inherently more challenging than from acetone-based systems [32]. Therefore, the type of solvent used in an adhesive formulation plays a critical role in determining its clinical performance by influencing both the extent of monomer conversion and the durability of the adhesive interface. During polymerization, the temperature attributable to the light device increases with the duration of polymerization or the level of irradiance, and this increased heat contributes to evaporation of the solvents contained in the adhesive [18,28]. Among high-power light devices, the Valo device is also known to cause the highest temperature increase [28]. Accordingly, it would be expected that due to the ethanol content and the shorter rubbing time, more residual solvent would remain in CSU, resulting in lower bond strength and DoC. However, it is interesting to note that while displaying a similar DoC to GBU, CSU showed a higher bond strength. A previous study reported that the solvent evaporation rate of CSU after 25 s of air application was 37%, while that of GBU was 94.2% [33]. This indicates that the solvent evaporation caused by the polymerization-related temperature increase after air application would be quite limited for GBU because the amount of residual solvent is significantly lower. It is important to recognize that the solvent content or the amount of residual solvent alone does not significantly impact the effectiveness of an adhesive. Additionally, the type and content of the adhesive are crucial factors in determining adhesive success.

In this case, however, it is clear that the amount of residual solvent in GBU would be lower than in CSU. Despite expectation, no difference in DoC among the adhesives was evident. This seemingly contradictory situation can be explained by considering that the ideal solvent concentration for optimal polymerization of adhesive resins would be higher than that for optimization of their physical properties [34].

After TC, the μTBS value of CSU decreased, whereas that of GBU did not change. Many previous studies have found that bond strength decreases after TC [35]. Thermal cycling is a well-established method for simulation of intraoral aging. Approximately 10,000 cycles are considered equivalent to one year of clinical service [36]. Thermal stresses may lead to microcrack formation and interfacial fatigue, resulting in degradation at the adhesive interface, and consequently a reduction in bond strength. [37]. However, GBU – containing solvents with a lower vapor pressure – forms a thinner film layer after polymerization [38]. In the present case, this may have prevented degradation caused by thermal stress.

This study has identified the most common type of adhesive failure. After repair of a fractured restoration, it is theoretically desirable for the repair bond strength to equal the original cohesive strength of the repaired restorative material. Here, the cohesive strength of the CAD-CAM resin composite was found to be significantly higher than the repair bond strength of the experimental groups. However, due to the advanced physical and mechanical properties of CAD-CAM resin composites [39], it is acceptable to have more adhesive failure than cohesive failure after repair.

In summary, while this in vitro study has shown that polymerization of adhesives at 1,400 mW/cm² irradiation for 10 s improved the repair bond strength, the results should be considered carefully. The use of various materials for repair involves complex processes that are difficult to fully evaluate [40]. The present analysis of the cohesive strength of CAD-CAM composite material before and after TC indicated that the

repair bond strength values in the experimental groups were significantly lower than the cohesive strength of CAD-CAM resin composite. Under current conditions, it is challenging to achieve a repair bond strength that is approximately equal to the cohesive strength of CAD-CAM resin composite. However, more in vitro and clinical studies should be conducted with different adhesives and restorative materials to determine clinically acceptable bond strengths and to establish a clinical acceptable repair process.

Increased exposure time and different levels of irradiance were shown to significantly affect the μTBS and DoC of universal adhesives. The 1,400 mW/cm²-10 s protocol led to significant improvements of μTBS and DoC for CSU in comparison to other groups. TC also decreased the repair bond strength of CSU. For improvement of adhesive performance, these results highlight the critical need for optimization of polymerization parameters.

Abbreviations

10-MDP: 10-methacryloyloxydecyl dihydrogen phosphate; 4-META: 4-methacryloyloxyethyl trimellitate anhydride; Bis-GMA: 2,2-bis[4-(2-hydroxy-3-methacryloyloxypropoxy) phenyl] propane; Bis-MEPP: bis-methacryloxyethoxy phenyl propane; CAD-CAM: computer-aided design-computer-aided manufacturing; CSU: Clearfil S^3 Bond Universal; DoC: in situ degree of conversion; GBU: Gluma Bond Universal; HEMA: 2-hydroxyethyl methacrylate; MPa: megapascal; SEM: scanning electron microscope; TC: thermal cycling; UDMA: urethane dimethacrylate; μTBS: micro-tensile bond strength

Ethics Statements

Not applicable

Conflicts of Interest

The authors have no financial interest in the companies whose materials were employed in this study.

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Author Contribution Statement

FB: conception, methodology, formal analysis and investigation, writing - original draft preparation, writing - review and editing, funding acquisition, resources; MK: conception, methodology, formal analysis and investigation, writing - original draft preparation, writing - review and editing, funding acquisition, resources. All authors read and approved the final version of the manuscript.

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Data Availability Statement

Data generated during the current study are available from the corresponding author on reasonable request.

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