Effect of Different Curing Times and Intensities on Microtensile Bond Strength of Bulk-Fill Resin Composite: An In Vitro Study

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Abstract

Aim: This in vitro study aimed to evaluate the effect of three curing times and intensities on the microtensile bond strength of bulk-fill resin composite and to assess the effect of thermocycling on the microtensile bond strength

Subjects and Methods: A total of 18 freshly extracted caries-free human permanent molars were selected for this study. Teeth fixed in acrylic resin blocks were mounted in an automated diamond saw. Occlusal surfaces were flattened to the level of the dentino-enamel junction. A specially designed stainless steel holder was fabricated to hold and stabilize the acrylic block and the tooth during bonding and resin composite application. The bonding agent was applied to dentin with micro brush for 20 seconds with gentle agitation and light-cured for 20 seconds. Moreover bulk-fill resin composite was applied on each surface. The teeth were used to obtain ninety dentin rods. The rods were divided into three equal groups (n=30), according to light curing intensity and time (I): Group (I_1): high intensity (2200 mw/cm^2) for one second, Group (I_2): high intensity (2200 mw/cm^2) for 3 seconds, and (I_3), low intensity (1200 mw/cm^2) for 20 seconds. Each group was further divided into two subgroups according to thermocycling (C): either subjected (C_1) or not (C_0). Each subgroup was subdivided according to storage time (T): (T_1) 24 hours, (T_2) 3 months, and (T_3) 6 months. Microtensile bond strength measurements were done using the universal testing machine.

Results: The highest microtensile bond strength of the used resin composite was for specimens cured with low intensity light (1200 mw/cm^2) for 20 seconds (I_3), followed by those cured with high intensity light for 3 seconds (I_2), then those cured with high intensity light (2200 mw/cm^2) for one second (I_1) respectively.

Conclusions: 1-The bonding quality of resin composite to dentin is the matter of adhesion protocol rather than the time of curing cycle. 2-The use of conventional protocol for light curing with low intensity provides superior bond strength of resin composite to dentin if compared to fast curing protocol. 3-Thermocycling adversely affects the microtensile bond strength of resin composite to tooth tissue irrespective of the bonding technique or the materials used. 4-Aging of resin composite may lead to decrease in microtensile bond strength of resin composite to dentin.

Keywords: Curing time, Curing intensity, Microtensile bond strength, Thermocycling.

Introduction

Resin composite restorative materials are commonly used for direct anterior and posterior restorations, because of their excellent esthetic properties, minimal coefficient of thermal expansion, and superior resistance to wear **(3)**. Usually, cavities are restored incrementally at a maximum depth of 2mm for each increment. The incremental packing allows efficient curing of the whole depth of the material and reduces polymerization shrinkage **(23)**. However, this technique consumes more time and increases the risk of air bubble entrapment, particularly when restoring deep cavities **(18)**.

In today's dentistry, there is a continuing pursuit to simplify and accelerate the clinical application of restorative materials **(3), (33)**. A new class of resin composite restorative materials called "bulk-fill resin composite" has been developed to facilitate restorative procedures by allowing the application of restorative material in thick increments up to 4-5 mm **(30)**. Because of changes in the filler content and organic matrix, bulk-fill resin composite restorative materials have a low modulus of elasticity and low polymerization stresses **(20)**.

Many factors, including the degree of conversion and depth of cure, influence the clinical performance of resin composites. Depth of cure (DOC) describes the amount of monomers converted to polymers within the resin composite material **(12)**. Several factors affect DOC, such as the percentage and type of fillers, the shade and thickness of the restoration, and the effectiveness of the light curing unit. **(21)**. Inadequate polymerization causes lower hardness and wear resistance, higher cytotoxicity, and increased marginal discoloration **(34), (16)**.

The light emitting diode (LED) curing unit is now the most widely used form of light curing units (LCUs) **(10), (16)**. Advances in light curing units have occurred in conjunction with improvements in resin composite formulations. One of these advances was the introduction of LCU with a high irradiation intensity ranging from 4000 mW/cm^2 to 5000 mW/cm^{2}in a very short time ranging from 1-3 seconds. **(27), (8)**.

Mechanical tests are usually implemented to assess the properties and predict the clinical performance of restorative materials **(6), (14)**. Many bond strength tests, such as tensile bond strength, shear bond strength, and push out tests, were traditionally used. These tests might be carried out on micro and macro scales. **(5)**. The microtensile bond strength (TBS) was widely used due to the enhanced distribution of stresses at the adhesive interface **(24)**. Therefore, this in vitro study aimed to evaluate the effect of different curing times and intensities on the microtensile bond strength of bulk-fill resin composite. The null hypothesis tested in this study was that there would be no difference in the microtensile bond strength of bulk-fill resin composite when using high intensity light curing (2200 mw) for short exposure time (1 second), high intensity light curing (2200 mw) for long exposure time (3 seconds), and Conventional light curing (1200 mw) for long exposure time for (20 seconds).

Materials and methods

The ethical aspects of this study were revised and approved by the Research Ethics Committee – Faculty of Dentistry – October 6 University in October 2021 (Approval No.RECO6U/10-2021).

A power analysis was designed to have adequate power to apply a statistical test of the null hypothesis that no difference would be found in the microtensile bond strength between the tested groups. By adopting an alpha level of (0.05), a beta of (0.2), i.e. power = 80%, and an effect size (f) of (0.637) calculated based on the results of **(13)**, the predicted sample size (n) was a total of (90) samples. G*Power version 3.1.9.7 was used to calculate sample size.

A total of 18 freshly extracted, caries-free human permanent molars were selected for this study. The teeth were extracted for periodontal purposes. Any tooth that had cavities, micro cracks, or other defects was discarded. The teeth were kept in distilled water at room temperature until they were used. The teeth were used to obtain ninety dentin rods. The rods were divided into three equal groups (n=30), according to light curing intensity and time(I): Group (I_1): high intensity (2200 mw/cm^2) for one second, Group (I_2): high intensity (2200 mw/cm^2) for 3 seconds, and Group (I_3): low intensity (1200 mw/cm^2) for 20 seconds. Each group was further divided into two subgroups according to thermocycling (C): either subjected (C_1) or not (C_0). Each subgroup was subdivided according to storage time (T): (T_1) 24 hours, (T_2) 3 months, and (T_3) 6 months.

A cylindrical Teflon mold (15-mm diameter and 40-mm height), with a corresponding metal ring with two opposing screws at its top was used to produce acrylic resin blocks. Teeth fixed in acrylic resin blocks were then mounted in an automated diamond saw (Isomet 4000, Buehler Ltd., Lake Bluff, IL, USA)**,** which was used for all sectioning procedures in this study. Occlusal surfaces were flattened to the level of the dentino-enamel junction under copious water coolant (Cool 2 water-soluble anticorrosive cooling lubricant, Buehler Ltd., Lake Bluff, IL, USA), with a concentration of 1:30, lubricant: water.

A specially designed stainless-steel holder was fabricated to hold and stabilize the acrylic block and the tooth during bonding and resin composite application. This specially designed device prevented the movement of both the acrylic block and the split Teflon ring which ensured proper completion of the bonding procedure steps. A dual cured universal bonding system (Futurabond

M^+,VOCO,CUXHAVN, GERMANY). The bonding agent was applied to dentin with a micro brush for 20 seconds with gentle agitation, air-thinned gently for 5 seconds and light-cured for 20 seconds with (I led, woodpecker, China; 1200 mw/cm^2**)**.A microhybrid resin composite (X-tra fil, VOCO, CUXHAVN, GERMANY).The resin composite was applied to the flat dentin surface and filled the internal surface of the Teflon mold as one bulk increment using gold plated composite applicator and light cured using a violet-blue LED curing unit (I led, Woodpecker, China; emission wavelength range: 420–480 nm). Three curing protocols were investigated: the protocol designated as "1-s" involved light-curing for 1 second with a radiant existence of 2200 mW/cm^2 and the protocol designated as "3-s" involved light-curing for 3 seconds with a radiant existence of 2200 mW/cm^2, whereas the protocol designated as "conventional" involved light-curing for 20 seconds with a radiant existence of 1200 mW/cm^2. The light curing intensity was checked every time before curing using a digital radiometer (LM-1, DTE, China).The materials used as well as their descriptions, principal components and manufacturers are listed in Table (1).

All samples were stored in distilled water at room temperature until microtensile bond strength testing. Half of the specimens in each subgroup were exposed to 2500 cycles between 5°C and 55°C with a dwell time of 30 seconds in each water bath and a 10 second transfer time from one bath to another using a thermocycling machine (SD Mechatronic Thermocycler, Germany).

The objective of longitudinal sectioning of restored teeth was to obtain resin composite-dentin beams of (1 mm x 1 mm) in area. Each beam was composed of resin composite and dentin, with adhesive at the interface. For the longitudinal sectioning to be perpendicular to the flat occlusal surface of restored teeth, a specially designed gripping attachment was used to hold acrylic blocks with mounted teeth firm in place, parallel to the sectioning direction, thus maintaining the perpendicular relation between the cutting disc and the occlusal surface. A metal house with two screws was used for firm attachment of acrylic blocks at the square base to ensure standard cutting in bucco-lingual and mesio-distal with 90° to each other, which is used to mount the attachment into the diamond saw machine. After mounting in the gripping attachment, restored teeth were serially sectioned, using a 0.3 mm thick diamond-coated disc (Buehler, IL, USA*)* under copious coolant. Serial sectioning was done in the bucco-lingual direction, then rotated 90°clockwise and sectioned in the mesio-distal direction. A final horizontal cut at the level of the cemento-enamel junction was done to obtain beams. The resultant beams were 1±0.1 mm in thick. A digital caliper (Total tools, Malaysia) was used to check the thickness and length of all beams.

Each beam was glued on Geraldeli’s jig by its ends using cyanoacrylate based glue (Akfix 705 fast adhesive, Turkey) away from the adhesive interface at least 1 mm, glue accelerator was used to accelerate hardening of the glue. The jig was in turn mounted into the universal testing machine

(Instron, MA, USA) with a load cell of 500 N. Tensile load was applied, at a cross-head speed of 0.5 mm/min, until bonding failure of the specimen occurred. Bond strength was calculated in Mega Pascal (Bluehill Lite software, Instron, MA, USA).

For statistical analysis, categorical data were presented as frequency and percentage values and were analyzed using Fisher's exact test. Numerical data were presented as mean and standard deviation (SD) values. They were explored for normality by checking the data distribution and using Shapiro-Wilk test. Data showed parametric distribution and were analyzed using one-way ANOVA followed by Turkey's post hoc test. The significance level was set at $p \leq 0.05$. Statistical analysis was performed with R statistical analysis software version 4.1.3 for Windows.

Results

Intergroup comparisons mean and standard deviation values of microtensile bond strength (MPa) for different curing intensities were presented in **Table (2)** and **Figure (1)**.

-For non-thermocycled specimens (C_0)

After 24 hours (T_1),the microtensile bond strength for the low intensity (1200 mw/cm^2) for 20 seconds specimens (I_3) was (30.12±6.61MPa), followed by high intensity (2200 mw/cm^2) for 3 seconds specimens (I_2) (28.73±3.72MPa), then high intensity (2200 mw/cm^2) for one second specimens (I_1) (25.20±1.50MPa) respectively. No significant difference between different groups was revealed (p=0.239).

After 3 months storage (T_2), the microtensile bond strength for low intensity (1200 mw/cm^2) for 20 seconds specimens (I_3) was (29.00±1.04MPa), followed by high intensity (2200 mw/cm^2) for 3 seconds specimens (I_2) (26.04±2.48MPa), then high intensity (2200 mw/cm^2) for one second specimens (I_1) (23.70±4.26MPa) respectively. A significant difference between different groups was apparent (p=0.042).

After 6 months storage (T_3), the microtensile bond strength for low intensity (1200 mw/cm^2)for 20 seconds specimens (I_3) was (25.20±1.50MPa), followed by high intensity (2200 mw/cm^2) for 3 seconds specimens (I_2) (20.12±1.96MPa), then high intensity (2200 mw/cm^2) for one second specimens (I_1) (15.48±0.88MPa) respectively. A significant difference between different groups was revealed (p<0.001).

-For thermocycled specimens (C_1):

After 24 hours (T_1), the microtensile bond strength for low intensity (1200 mw/cm^2) for 20 seconds specimens (I_3) specimens was (25.07±3.06MPa), followed by high intensity (2200 mw/cm^2) for one second specimens (I_1) (16.35±0.66MPa), then high intensity (2200 mw/cm^2) for 3 seconds specimens (I_2) (16.35±0.11MPa) respectively. A significant difference between different groups was apparent (p=<0.001).

After 3 months storage (T_2), the microtensile bond strength for low intensity (1200 mw/cm^2) for 20 seconds specimens (I_3) was (23.27±4.34MPa), followed by high intensity (2200 mw/cm^2) for 3 seconds specimens (I_2) (15.78±2.70MPa), then high intensity (2200 mw/cm^2) for one second specimens (I_1) (14.10±2.54MPa) respectively. A significant difference between different groups was revealed ($p=0.002$).

After 6 months storage (T_3), the microtensile bond strength for low intensity (1200 mw/cm^2) for 20 seconds specimens (I_3) was (16.27±2.17MPa), followed by high intensity (2200 mw/cm^2) for 3 seconds specimens (I_2) (14.46±2.84MPa), then high intensity (2200 mw/cm^2) for one second specimens (I_1) (5.35±1.59MPa) respectively. A significant difference between different groups was revealed ($p<0.001$).

Discussion

The advancement of resin composite restorations, universal bonding agents, and high-intensity light-curing devices are just a few examples of how adhesive dentistry materials and methods have changed over time in a trend that is always toward the simplification of restorative procedures. The benefits provided by these advances go beyond merely raising the cost-effectiveness of restorative care; they also result in efficient processes that preserve time for both patients and operators and reduce the risk of iatrogenic errors **(17)**.

By reducing the amount of time and steps needed for the restorative procedures, current research in dentin adhesives has focused on making it easier to apply bonding agents. Since the self-etch approach offers dependable adhesion to dentin, it was used in the current study's universal adhesive one step system. The used adhesive system contains highly functionalized SiO2 nano particles (20 nm), which aid in the cross-linking of the resin's constituent parts, improve the film-building capabilities, and reinforce the hybrid layer for long-lasting high bond strength. Additionally, the manufacturer stated that the acidity (pH = 2), which was considered a mild self-etch adhesive, allowed the adhesive to interact superficially with dentin, dissolve the smear layer, and penetrate it to form a more uniform and stable, resin-infiltrated hybrid layer, can provide a suitable bond strength to wet and dry dentin with secure adhesion, reduced postoperative sensitivity, and appropriate marginal integrity **(2) (22), (32)**.

Bulk-fill resin composite have been developed to make it simpler to place direct resin composites in deep cavities, save time, and reduce the chance of errors. This is because adding more increments to deep cavities increases the likelihood of incorporating voids and porosity, which could weaken the bond strength. Bulk-fill resin composites demonstrated sufficient dentin bond strength and can be used as the preferred restorative material in posterior teeth. They are also appropriate for the bulk technique because it enables the application of increments of up to 4 mm in a single step while preserving the mechanical properties, degree of conversion, and polymerization shrinkage stresses **(15), (19**).

A recent ultra high intensity LED curing unit was released with curing times of 1-3 seconds and an intensity of 4000–5000 mW/cm2. Because it requires less time to work in the clinic due to its fast time of curing, this LED is advantageous to both the operator and the patient **(8)**. In the current study, a violet-blue LED curing unit (I led, Woodpecker, China; emission wavelength range: 320-480 nm) was used with three different curing protocols to light cure the resin composite.

Distilled water was used in the present study for storage of the specimens after light curing, for 24 hours, 3 and 6 months respectively. Water storage is one of the most commonly used artificial aging techniques *because it* is a relatively simple and economical method. The bonded specimens were stored in distilled water at 37°C **(1), (31)**.

Thermocycling is meant to mimic the thermal stress that the restorative materials and teeth would be exposed to by consuming drinks and food, which would affect bond durability and occasionally lead to failure of restoration, and to provide the specimens with years of ageing in a short period of time. Many investigations have been conducted to evaluate the temperature variations in the mouth when drinking hot and cold beverages. When reviewing the literature, most investigators apply thermal cycling of specimens between 5°C and 55°C with a dwell time of 30 seconds and 2500 cycles, which is equivalent to 3 months physiological ageing in the oral environment and is adequate to cover oral temperature fluctuations **(7)**.

The microtensile bond strength (TBS) test was chosen for the current study because it has been widely used in numerous bond strength testing studies, making it one of the most common and adaptable tests for evaluating the bonding performance of materials in vitro. This testing approach allows for a greater number of specimens per tooth, increasing test accuracy. It also has a lower coefficient of variation and a reduced probability of cohesive fracture along dentin. It enables for the evaluation of resin adhesive performances to drilled carious or sclerotic dentin, as well as the regional bond strengths for different portions of the cavity. Furthermore, the opportunity to compare the long-term durability of resin adhesion at various regions of the cavity walls on teeth extracted at different periods following the placement of bonded restorations **(5), (24), (28)**.

In the present study, the highest microtensile bond strength of the resin composite was for specimens cured with low intensity light (1200 mw/cm^2) for 20 seconds (I_3), followed by those cured with high intensity light for 3 seconds (I_2), then those cured with high intensity light (2200 mw/cm^2) for 1 second (I_1) respectively**.** This might be due to the effect of percentage of resins monomers within the composite during polymerization as it may not be cured well if the light intensity or time of curing was not sufficient. Therefore the longer curing time irrespective of the light intensity may result in greater mechanical strength and other properties as a high microtensile bond strength **Harahap et al. (9)**. These results were in agreement with the results of **Lee et al. (11)**, who suggested that extended curing time can improve different mechanical property of bulk-fill resin composite. Moreover, **Makhdoom et al. (13)**, found that the degree of resin polymerization and high bond strength have a close relationship with the duration of light curing time rather than with the intensity of the emitted light. Although the advantage of the LED curing unit lies in having a high intensity of 2500 mW/cm^2, with a curing time ranged from 1–3 seconds, there is no much research showing the success of the tool in the complete polymerization of resins composite.

All of light cured resin composite specimens had higher microtensile bond strength in non-thermocycled specimens than thermocycled specimens, this might be due to that the artificial aging effect induced by thermocycling occurs in two ways: First; hot water may accelerate hydrolysis of the interface components, and subsequent uptake of water and extraction of breakdown products or poorly polymerized resin oligmers (diffusion dependent hydrolysis and elusion). Second, stresses induced by repetitive differential thermal changes (contraction/expansion stresses). These stresses may induce cracks that propagate along the bonded interfaces, and once a gap is created, changing gap dimensions can cause in and out flow of oral fluids **Szczesio-Wlodarczyk et al. (29)**. These results were in agreement with **Souza et al. (26), Osman. (17)**, who reported deterioration of bond strength after thermocycling.

The results of the present study showed that the microtensile bond strength values were adversely affected by water storage. This might be due to the biodegradation process of resin-based materials over time, where the daily rate of consumption of aqueous solutions drastically affects the µTBS of the resin-based materials. The bond strength decreased gradually with more aging of the resin composite in distilled water storage media. Interface components can be degraded by hydrolysis and water may infiltrate, resulting in the weakening of the polymeric matrix, by swelling and reduction of the frictional forces between the polymer chains, reducing the mechanical properties, and consequently the bonding interface integrity. These results in agreement with other studies that showed a significant decrease in the bond strength after water storage **Souza et al. (26), Craciun et al.(4), Selim et al. (25)**.

Conclusions:

Under the conditions of the present investigation the following conclusions could be drawn:

1. The use of conventional protocol for light curing with low intensity provides superior bond strength of resin composite to dentin if compared to fast curing protocol.

2. The bonding quality of resin composite to dentin is the matter of adhesion protocol rather than the time of curing cycle.
3. Thermocycling adversely affect the microtensile bond strength of resin composite to tooth tissue irrespective of the bonding technique or the materials used.
4. Aging of resin composite may lead to decrease in microtensile bond strength of resin composite to dentin.

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Table (1): Materials' specification, composition, manufacturer, and lot number

Material	Specification	Composition	Manufacturer	Lot number
Futurabond M^+	Dual cured universalbonding	1- 2-hydroxyethyl 2- 2-methacrylate (10–25%),	VOCO, CUXHAVN	2031283

	system	3- BIS-GMA (10–25%), 4- Ethanol (10–25%), 5- Acidic adhesive monomer (2.5–5%), 6- Urethanedimethacrylate (2.5–5% catalyst, 7- Pyrogenic silicic acids, catalyst.	GERMANY *www.voco.com*	
X-tra fil	Microhybrid resin composite (universal shade)	1- Bisphenol A glycidyal dimethacrylate 2- Urethane dimethacrylate, 3- Triethylene glycol, dimethacrylate. 4- Containing filers; Barium-boron alumino-silicate glass (2-3 µm) 86% by weight,.	VOCO, CUXHAVN GERMANY *www.voco.com*	2149109

Table (2): Intergroup comparisons mean and standard deviation values of microtensile bond strength (MPa) for different curing intensities.

Thermocyling	**Time**	**Microtensile bond strength (MPa) (mean±SD)**			**p-value**
		I_1	**I_2**	**I_3**	
C_0	**T_1**	25.20 ± 1.50^{A}	28.73 ± 3.72^{A}	30.12 ± 6.61^{A}	**0.239ns**
	T_2	23.70 ± 4.26^{B}	26.04 ± 2.48^{AB}	29.00 ± 1.04^{A}	**0.042***
	T_3	15.48 ± 0.88^{C}	20.12 ± 1.96^{B}	25.20 ± 1.50^{A}	**<0.001***
C_1	**T_1**	16.35 ± 0.66^{B}	16.35 ± 0.11^{B}	25.07 ± 3.06^{A}	**<0.001***
	T_2	14.10 ± 2.54^{B}	15.78 ± 2.70^{B}	23.27 ± 4.34^{A}	**0.002***
	T_3	5.35 ± 1.59^{B}	14.46 ± 2.84^{A}	16.27 ± 2.17^{A}	**<0.001***

Different superscript letters indicate a statistically significant difference within the same horizontal row *; significant ($p \le 0.05$) ns; non-significant (p>0.05).

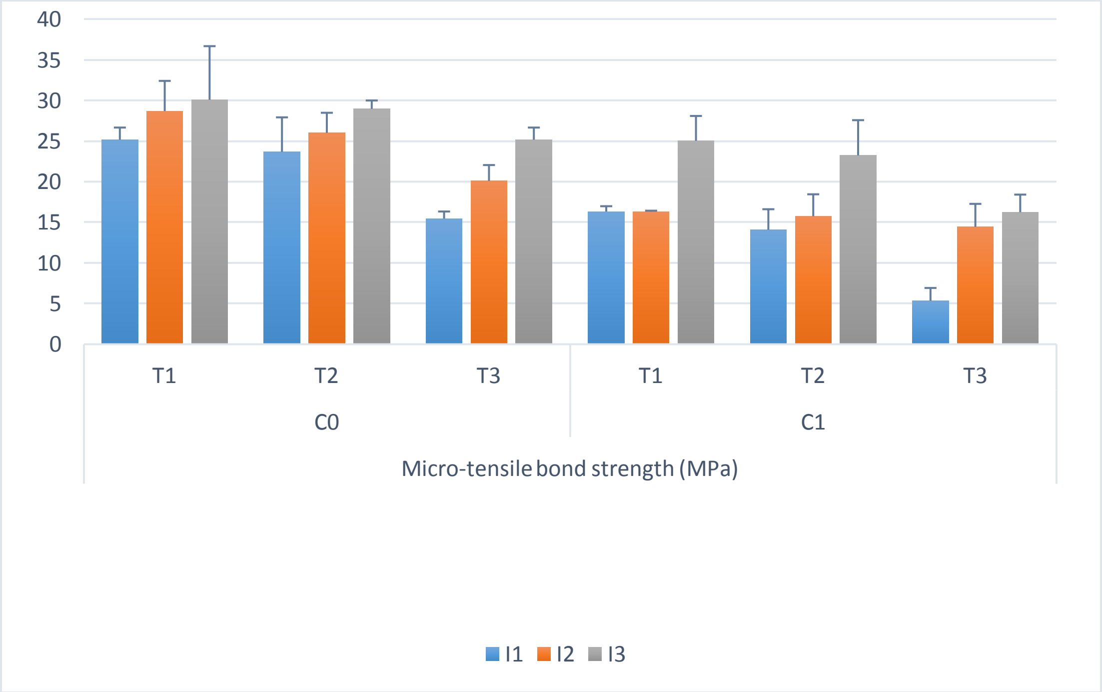

Figure (1) Bar chart showing average microtensile bond strength (MPa) for specimens in different curing times and intensities with /without thermocycling and different times of storage.