

Original Article

Effect of Shelf Life of Light Cure Resin Cement on the Shear Bond Strength of IPS EMAX PRESS Ceramic: (An *In vitro* Study)

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ABSTRACT

Background: Achieving durable bonding for all ceramic restorations will affect its long-term survival in the oral cavity. Dentists may use resin cement after its shelf lifetime, especially in low socioeconomic conditions, which may affect the success and longevity of restorations. The purpose of this *in vitro* study was to evaluate how the duration of shelf life impacts the shear bond strength (SBS) of lithium disilicate ceramics using light-cured resin cement.

Materials and Methods: An *in vitro* comparative study investigated the impact of resin cement expiry on the SBS of pressed lithium disilicate ceramics. Twenty-four IPS e.max Press discs (4 mm × 2 mm) were randomly divided into two groups: Group I used unexpired light-cured resin cement, and Group II used expired cement. Each disc was bonded to enamel surfaces from sectioned maxillary incisors to create test specimens. Following thermocycling, samples were subjected to a shear force using a universal testing machine until debonding occurred, and the failure load was recorded. Data normality was confirmed with the Shapiro-Wilk test, mean SBS values were compared through independent *t*-test, and failure modes were assessed using the Chi-square test with Monte Carlo correction at a significance level of $P < 0.05$.

Results: Group I exhibited a higher SBS with a mean value of 24.98 ± 4.01 MPa compared to Group II with a mean value of 20.39 ± 2.72 MPa, with a mean $P = 0.008$.

Conclusion: Expiry date affected the SBS of light-cured resin cement to lithium disilicate ceramic materials. But still the recorded value is higher than the recommended clinical accepted value.

Key Words: Ceramics, dental bonding, dental cements, *in vitro* techniques, shear strength

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INTRODUCTION

In recent times, advancements in technology and the progress of ceramics fabrication, such as CAD/CAM technology, intraoral scanners, and 3D printing, have given rise to heightened aesthetic expectations from both patients and dentists. Ceramic materials are widely embraced due to their natural appearance, biocompatibility, chemical stability, high compressive

resistance, and a thermal expansion closely resembling tooth structure.^[1,2] Various commercially available all-ceramic systems have been introduced for crafting complete coverage crowns and veneers. Despite significant strides in ceramic dentistry, achieving an esthetically pleasing restoration that closely matches neighboring teeth continues to pose challenges.^[3]

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A critical aspect of concern lies in the success of veneers, hinging on the bonding strength and durability of the tooth surface, resin cement, and ceramic components.^[4] The effectiveness of veneers is intricately tied to the physical and mechanical properties of adhesive materials, the polymerization mechanism, and the curing mode. In general, resin cement plays a crucial role in achieving both robust bonding strength and minimal film thickness, the latter being essential for optimal esthetics.^[5]

The degradation of resin luting cement involves a multifaceted process. Two key processes are evident: intraoral degradation, which encompasses mechanical, physical, or chemical factors, and extraoral degradation, resulting from the material's storage conditions and shelf life. Numerous dental materials are susceptible to degradation and come with specific storage prerequisites to uphold their optimal properties and extend their shelf life.^[6]

Dental materials are often stored unused in dental clinics and should be stored in accordance with manufacturer-specific guidelines. During this period, it is crucial to ensure that the material's ingredients do not undergo separation, evaporation, reactions with each other, degradation, or any alteration.^[7]

To preserve their properties, it is essential to store resin cements and composites appropriately.^[8] The recommended storage temperature typically falls within the range of 4°C to 20°C. However, it is important to note that the storage conditions for resin composites may vary depending on the geographical and climatic conditions of the country, taking into consideration factors such as sun exposure and humidity.

Selecting an appropriate luting cement is crucial, as the ceramic-tooth bonding process depends on the cement's adhesion to both the ceramic substrate and tooth structures, including enamel and dentin.^[9,10] Among various techniques for evaluating bond strength, the shear bond and microtensile tests are most commonly employed.^[11] The shear bond test was selected for this study due to its proven reliability in prior research.^[12-14]

Hondrum and Fernandez^[15] reported that chemically cured dental resins showed reduced mechanical strength and longer working and setting times during the first four years of storage, after which their properties stabilized. In contrast, light-cured resins maintained consistent properties throughout seven years, regardless of storage conditions.

Gielzak *et al.*^[16] conducted a study examining the effect of storage temperature on selected strength parameters of dual-cured composite cements. Their findings revealed that the strength properties (three-point flexural strength and diametral tensile strength) were not dependent on storage temperature in the range of 8°C–35°C.

Fallo *et al.*^[8] conducted a study to examine the impact of improper storage on the polymerization, handling, and appearance of visible light-cured composite resin and resin-modified glass ionomer materials. Their findings revealed that these materials showed no significant changes in properties and that their clinical performance remained unaffected when stored within a temperature range of 20°F to 112°F for 12 months.

Turini *et al.*^[17] investigated the effect of shelf life on the bond strength and microhardness of self-adhesive resin cements and found that expiration after 6 months or 1 year, when stored under manufacturer-recommended conditions, did not affect either property.

The expiration date of resin composites and cements is an important consideration, as using them beyond this date can alter their properties and potentially cause clinical issues such as debonding, restoration fractures, or discoloration. Although some practitioners may use materials past the manufacturer's recommended date, it remains unclear whether they retain sufficient performance or should be discarded.

This study aimed to assess how the expiration of resin cement affects the shear bond strength (SBS) of pressed lithium disilicate ceramics (IPS e.max Press). The null hypothesis proposed that no significant difference in bond strength would exist between expired and unexpired light-cured resin cements.

MATERIALS AND METHODS

An *in vitro* comparative experiment was conducted to assess how the expiration of light-cured resin cement affects the SBS of pressed lithium disilicate ceramics. The study was approved by the Institutional Review Board of the Faculty of Dentistry, Alexandria University (IRB No. 00010556; IORG No. 0008839; approval No. 0853-1/2024). All procedures were conducted in accordance with the Declaration of Helsinki and the ethical guidelines of the Research Ethics Committee, Faculty of Dentistry, Alexandria University. No human participants were involved in this study.

Sample size was based on a 95% confidence level to detect differences in bond strength in respect to the resin luting cements. The minimum required sample size was calculated to be 9 specimens per group, increased to 10 to make up for laboratory processing errors. The total required sample size = number of groups \times number per group = $2 \times 10 = 20$ specimens.^[18]

Hopeless mobile maxillary central incisors (Grade III or IV mobility) were collected from diabetic patients in the department of surgery, Faculty of Dentistry, Alexandria University. A total of 24 human sound freshly extracted upper central incisors were selected for this study. Initially, teeth were mechanically cleaned by a periodontal hand scaler to remove any debris, calculus deposits or remaining soft tissues. After mechanical debridement of the teeth, the surfaces were cleaned with pumice water slurry. For sterilization of the extracted teeth, they were subsequently kept in de-ionized water, 0.2% glutaraldehyde, which was effectively enough to destroy all kinds of microorganisms. Finally, thymol was added to water to inhibit bacterial growth.^[19]

All teeth were labially prepared to achieve a flat enamel surface using a depth-limiting bur (0.5 mm). To standardize the enamel surface while remaining within the enamel layer, the labial surfaces of the crowns were reduced using a depth-controlled approach. Pilot sectioning of two nonstudy incisors revealed an average labial enamel thickness of approximately 0.8 mm; therefore, a target removal depth of 0.5 mm was selected to ensure that preparation remained within the enamel. The root portions of all the teeth were cut off by a diamond disc. Each specimen was embedded in a self-curing acrylic resin block (Acrostone, Cairo, Egypt) fabricated using a metallic mold in which the mixed resin dough was placed. The cut tooth specimen was embedded and centralized into the acrylic resin exposing the labial surface only until polymerization occurred. A clean glass slab was placed on top of the prepared labial surface of the specimen to ensure a flat surface for the specimen. After complete polymerization, the cut labial surface was then smoothed using a smooth sandpaper disc (600–1000 grit, SiC (Struers, France) under running water to obtain a flat surface for bonding procedures. The 24 specimens were randomly divided into two main Groups: I before cement expiry and II 6 months after cement expiry ($n = 12$).

Discs (4 mm in diameter \times 2 mm in thickness) were first digitally designed using computer-aided design software (Auto CAD; Autodesk Inc). The specimens

were dry-milled in CAD-CAM wax blanks (Ceramill white wax; Amman Girrbach AG) using a milling machine (Ceramill Motion 2; Amann Girrbach AG). The sprued wax discs were then invested using phosphate-bonded investment material (Bego USA, Boston, United States), which was mixed according to the manufacturer's instructions. Following the investment material setting time, the investment ring was then placed in a burn-out furnace at 850°C for 60 min. The heat pressing process of IPS e.max press was performed in a pressing furnace (Programat Furnace EP 3010; Ivoclar AG) at 925°C for 25 min, following the manufacturer's instructions. The discs were finished using silicon carbide paper under cooling water, and then the dimensions of the specimens were checked using a digital caliper. All the specimens were cleaned ultrasonically in distilled water for 10 min, and then randomly divided into two groups as mentioned before. All the discs were etched by hydrofluoric acid 9% (Ultradent, Manly, Australia) for 60 s.^[20] After etching, the etched surface was rinsed thoroughly with a simultaneous mixture of air and water. This step is essential to remove any remaining hydrofluoric acid and etching debris, and then dried. Followed by application of silane coupling agent Monobond S (Ivoclar Vivadent, Schaan, Liechtenstein) using a brush for 60 s, then gently air-dried with a dry, oil-free air stream for a few seconds.

Variolink esthetic light cure resin cement (Ivoclar Vivadent, Schaan, Liechtenstein) was used in this study, 2 syringes, one before its expiry and the other after the expiry date by 6 months; both were stored in a refrigerator at 4°C.

The exposed flat enamel surface of each specimen was etched using 37% phosphoric acid (Ivoclar Vivadent, Schaan, Liechtenstein) for 20 s, followed by rinsing with water for 10 seconds, then air-dried by air/water spray syringe. The resin cement was dispensed from the syringe directly on the prepared labial surface of each tooth. Each ceramic disc was held by a specially modified serrated tweezer and placed onto the demarcated area of the specimen (cement area).

Following the application of manual finger pressure to accurately position the disc, all specimens were seated by a single, trained operator. To ensure consistency, the operator underwent a pre-test calibration procedure, during which finger pressure was practiced on a balance to approximate the intended load, excess cement was removed with an explorer, and glycerin gel was applied to the tooth-ceramic interface to

prevent oxygen inhibition in surface layer of the cement. Each specimen was then placed under the static load device of 2.0 kg for 5 min to standardize the exerted pressure. Specimens were exposed to the polymerization light of 800 mW/cm² for 40 s from 4 straight opposite directions using the LED visible light curing unit (LITEX 695 DENTAMERICA, USA.).

Specimens were subsequently subjected to thermocycling for 5000 cycles in water baths alternating between 5°C and 55°C, with a dwell time of 20 s in each bath and a 10-second transfer interval. This protocol conducted at a rate of 300 cycles per day until completion to simulate approximately six months of clinical service.^[21,22]

Shear bond testing was performed to debond the ceramic discs from the prepared labial surface using the universal testing machine (5ST; Tinius Olsen, England) at a crosshead speed of 1 mm/min. A chisel-shaped blade was used to exert the load at the bonded surface between the prepared enamel and ceramic disc surfaces. The load at which the bond failed, and the ceramic discs were separated from the enamel surface, were recorded in Newtons through a digital monitor.

The SBS (MPa) was calculated according to the following equation:

Shear bond = fracture load (N)/surface area of the disc (mm²)

Where area of the disc = πr^2

A specimen from each group was examined under a scanning electron microscope (SEM) (Jeol JSM-IT200; Jeol Ltd., Tokyo, Japan). Each specimen was coated with gold sputter coating in a machine before SEM examination. After gold coating, images were captured at magnifications of ($\times 2000$) with an accelerating voltage of 15 KV to examine the surface morphology of each disc. The surfaces of the discs after debonding were examined under a stereomicroscope at $\times 2.5$ (SZ1145TR Olympus; Japan 1990) by using a software (Toup view, version 3.7). Failure mode was classified as follows: cohesive when the failure occurs in the tooth surface or within the ceramic disc, adhesive when most of the resin cement is noticed on the tooth surface, and mixed failure when some resin cement is noticed together with tooth structure.

Statistical analysis

Data were analyzed using IBM SPSS Statistics Version 26.0 (IBM Corp., Armonk, NY, USA).

Normality was evaluated through descriptive statistics, normality plots, and the Shapiro-Wilk test. The independent samples *t*-test compared mean SBS between unexpired and expired cement groups, whereas the Chi-square test with Monte Carlo correction assessed differences in failure modes. Results were presented as mean \pm standard deviation, with statistical significance set at $P < 0.05$.

RESULTS

For SBS, quantitative data were described using mean and standard deviation. The mean SBS in MPa are presented in Table 1 and Figure 1a, showing the highest SBS for the unexpired Group I (24.98 ± 4.01) and for expired Group II (20.39 ± 2.72). Group I showed a statistically significant higher SBS value compared to Group II.

For the failure mode, no statistical significant difference was noted between the two tested groups as shown in Table 2 and Figure 1b using the Chi-square test. Pictures taken under a stereomicroscope illustrate the mode of failure in both tested groups, as shown in Figures 2 and 3.

For SEM analysis after debonding, mixed and adhesive failure were the most prevalent failure modes reported in both groups, also remnants of resin cement penetrating the tooth surface were noticed in a similar manner in both tested groups as shown in Figure 4.

DISCUSSION

This *in vitro* study investigated the effect of resin cement expiration on the SBS of pressed lithium disilicate ceramics, demonstrating that expired

Table 1: Shear bond strength in the two study groups

Tested groups	Mean \pm SD (MPa)	95% CI	P
Unexpired group (I)	24.98 ± 4.01	22.11–27.84	0.008*
Expired group (II)	20.39 ± 2.72	18.45–22.33	
Difference	4.58 ± 4.38	1.37–7.80	

*Statistically significant at $P < 0.05$. SD: Standard deviation; CI: Confidence interval

Table 2: Failure mode in the two study groups

Tested groups	Unexpired, n (%)	Expired, n (%)	P
Cohesive	2 (16.66)	0	0.13
Adhesive	4 (33.3)	7 (58.3)	
Mixed	6 (50)	5 (41.6)	

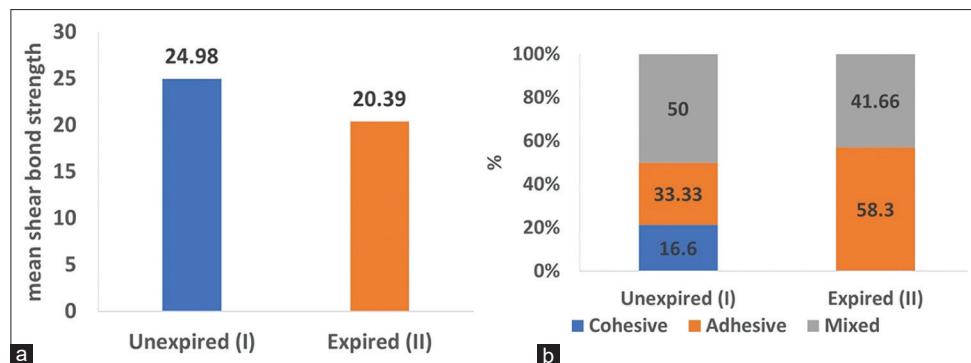


Figure 1: (a) Graphical presentation for shear bond strength values for the tested groups, (b) Mode of failure graphical presentation.



Figure 2: (a) Showing cohesive failure within the tooth itself. (b) Adhesive failure. (c) Mixed failure for Group I specimens.

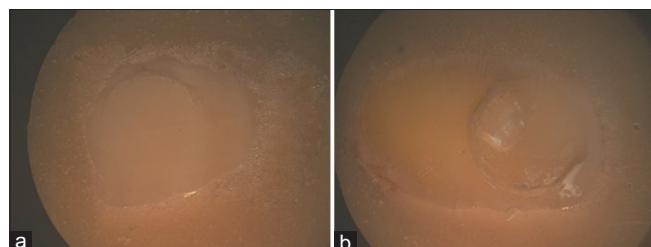


Figure 3: (a) Showing adhesive failure for Group II specimens. (b) Mixed failure for Group II specimens.

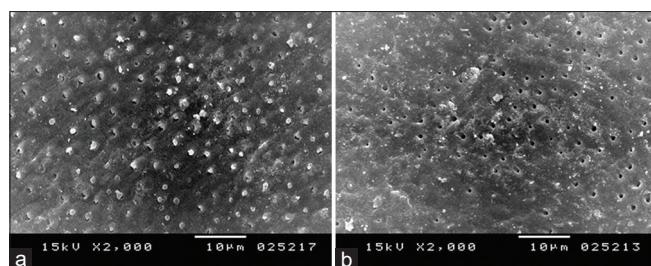


Figure 4: Scanning electron microscope image showing remnants of resin cement on the tooth surface in a similar pattern. (a) More resin cement remnants were noticed in Group I. (b) Less apparent resin cement remnants in Group II.

cement significantly reduced bond strength compared to unexpired cement. Such *in vitro* studies are important for analyzing restoration failures, including fractures of restorations or tooth structures, and offer advantages over clinical studies in terms of cost, reproducibility, and control over experimental variables.^[13,14] However, to generate results that are

meaningful and translatable to clinical practice, the experimental setup must closely replicate real-world conditions, ensuring that findings are both relevant and applicable to actual restorative procedures. The null hypothesis was rejected as the unexpired group exhibited higher SBS than the expired group.

IPS e.max Press, a heat-pressable lithium disilicate glass-ceramic, was chosen for this study due to its excellent esthetics, high translucency, good wear resistance, and an internal surface that can be etched for bonding. According to the manufacturer, the material contains 60%–80% lithium disilicate crystals embedded in the glass matrix. These crystals help stop cracks from spreading, which contributes to the material's overall strength and durability.^[23,24]

It is widely acknowledged that dental ceramic materials, once placed in the oral cavity, face a challenging environment. Ide *et al.*^[25] and Wegner *et al.*^[26] have investigated the impact of thermocycling on both the overall strength and the bond strength of ceramics. In the present study, thermocycling was conducted to replicate intraoral temperature fluctuations experienced during routine activities such as eating and drinking. The specimens underwent 5000 cycles of thermocycling at temperatures of 5°C and 55°C, which corresponds to approximately one year of clinical service.^[26,27]

Extracted teeth were used as the substrate in this study because their elasticity, thermal conductivity,

bonding behavior, and strength closely mimic clinical conditions, offering a more realistic simulation than plastic, resin, or metal alternatives. Many studies have shown that natural teeth are preferred for testing, as adhesive bonding procedures in practice are typically performed on all-ceramic restorations.^[28,29]

In the shear bond testing of all-ceramic specimens, Variolink Esthetic resin cement was employed following the manufacturer's recommendations. This light-cured resin cement, chosen for its adhesive properties, is the preferred material for luting indirect tooth-colored restorations <2 mm thick.^[8]

The study results revealed a statistically significant difference in SBS between Group I and Group II. This observation is consistent with the expectation that the unexpired resin cement (Group I) demonstrated higher values due to its composition remaining unaffected by degradation or chemical changes that may have occurred in the second group. It is noteworthy that resin composites vary in the amount of photo initiator they contain, and all photo initiators degrade over time.^[15] The catalyst peroxide within the paste is crucial for longevity, and some formulations are better stabilized than others.^[8]

The SBS values obtained in this study align with reported values in literature, falling within the range of 20–31 MPa observed in many trials. This consistency may be attributed to similarities in the test settings and methodologies employed across studies.^[14,30,31]

Despite the lower values recorded for the expired group (II), with values at 20.39 ± 2.72 MPa, these results still fall within the clinically acceptable range reported in literature.^[14,20,30] This suggests that there is no severe deterioration in the physical and chemical properties of the expired light-cured resin cement. The stability exhibited by light-cured resin cement is noteworthy, especially when compared to dual and chemical cure resins that tend to be more sensitive to storage conditions and consequently have a shorter shelf life. The presence of an unstable benzoyl peroxide initiator in the curing system of these resins makes them more susceptible to degradation.^[15] Proper storage in dark conditions and under refrigeration significantly prolongs the shelf life of resin cement by slowing down the decomposition of the photo initiator.^[32]

Ozer *et al.* investigated the bonding performance of three novel self-adhesive resin cements to human dentin under two different storage conditions. Their findings demonstrated a significant reduction in bond

strength following storage at room temperature, indicating that adherence to the manufacturer's recommended storage conditions may help preserve the cement's effectiveness. This supports the high bond strength values observed in the unexpired groups.^[32]

For the failure mode analysis, the cohesive failure within the tooth was noted only in the unexpired group that proves the better bond and union that occurs upon comparing to the unexpired group, still the mixed failure is the highest among both groups. No difference also, was also detected for SEM analysis for the tooth surface after debonding, as similar penetration of the cement on the tooth surface was noted. Some studies showed that proper storage and refrigeration may prolong the shelf life of composites and luting agents for a period ranging from 6 to 12 months;^[8,15,33] Whereas other studies found that refrigeration of resin-composites might have a deleterious effect on the degree of conversion and microhardness of the tested composite restorative materials with different matrix systems.^[34]

The shelf life of a material refers to the duration, starting from the date of manufacturing, during which the material maintains the physical and mechanical properties essential for fulfilling its intended purpose. Recently, the FDA has reported that many prescription drugs may remain effective beyond their expiration dates.^[35] In the context of dental products, alterations in properties over a few years may not be immediately clinically apparent but could potentially impact the longevity of restorations.^[15,32]

Limitations of this study

EDX and X-ray diffraction may be employed on both expired and unexpired resin cement to detect any noticeable changes to the materials. Furthermore, assessment of the color stability of expired light-cured resin cement is of great importance to be studied. The present study utilized a single type of resin cement.

CONCLUSION

1. Unexpired group exhibited a higher SBS than the expired groups
2. The unexpired group exhibited a clinically acceptable bond strength exceeding 20 MPa.

Limitations of the study

1. Different types of resin cements should be included and tested

2. Elemental analysis, XRD and more chemical tests should be done to evaluate the changes in the composition of the resin cements before and after expiry.

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Conflicts of interest

The authors of this manuscript declare that they have no conflicts of interest, real or perceived, financial or nonfinancial in this article.

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