

Original Article

Comparison of Shear Bond Strength of Silorane and Nanohybrid Composite Resins to Calcium Enriched Mixture Cement in Different Time Periods

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ARTICLE INFO

Article History:

Received: 19 October 2017

Accepted: 21 January 2018

Key words:

Calcium enriched mixture

Nanohybrid

Shear bond strength

Silorane

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Abstract

Statement of problem: It is crucial to select the best dental material for restoration of teeth that undergo vital pulp therapy after endodontic treatment.

Objectives: Adhesion of composite resin and endodontic pulp capping material affect the outcome of treatment. The aim of the present study is to evaluate the adhesion of two different composite resins to calcium enriched mixture (CEM).

Materials and Methods: We prepared a total of 60 cylindrical acrylic blocks that contained a central hole (4 mm diameter and 2 mm height). CEM cement was mixed according to the manufacturer's instructions and introduced into the holes. We stored 30 specimens for 3 days and the remaining 30 specimens were stored for 7 days at 37°C. The specimens were further divided into 2 subgroups based on the composite resins that we used. One subgroup of specimens was restored by Z350 (3M, ESP, St. Paul, MN, USA) nanohybrid and the other restored by P90 (3M, ESP, St. Paul, MN, USA) silorane based composite. Shear bond strengths were measured by a universal testing machine. Failure modes of the samples were evaluated under a stereomicroscope.

Results: At day 3, P90 had significantly higher shear bond strength than Z350 (P=0.001). On day 7, Z350 had significantly higher shear bond strength compared to P90 (P=0.004).

Conclusions: Within the limits of the present study, the best results of P90 silorane based composite filling after vital pulp therapy with CEM cement biomaterial were obtained after 3 days, whereas the Z350 nanohybrid composite showed better results on day 7.

Cite this article as: Koohpeima F, Sobhnamayan F, Esfandiari M, Siabani S. Comparison of Shear Bond Strength of Silorane and Nanohybrid Composite Resins to Calcium Enriched Mixture Cement in Different Time Periods . J Dent Biomater, 2018;5(2):559-566.

Introduction

Composite resins are extensively used in dentistry for esthetic areas. Because of favorable esthetic results of composite restorations, they are popular among both clinicians and patients. Dental composite resins have improved since their introduction. However, despite the improvements, some of the shortcomings of composite materials have not been overcome. Numerous studies have emphasized decreasing polymerization shrinkage, which is one of the main shortcomings of the resin composites. Silorane based composite, a low-shrinkage tooth-colored restorative material, has been introduced to the market. Silorane is a hybrid biomaterial of oxirane and siloxane [1]. The functional groups of oxirane are responsible for lower shrinkage while siloxane specify the highly hydrophobic nature of the siloranes, compared to methacrylate-based composites. While methacrylate-based composites polymerize via a free-radical mechanism, Oxiranes, polymerize by a cationic ring-opening mechanism. They also show better performance in maintaining the bond interface because of their highly hydrophobic properties [2]. Developments in the manufacture of resin composites led to production of nanocomposites with better mechanical and handling characteristics, higher aesthetic aspects, and decreased polymerization shrinkage. The use of nanotechnology in production of nanocomposites has resulted in higher polishability and polish retention, high translucency, proper maintenance of physical properties, and high wear resistance [3, 4]. In the last few years, one of the most important improvements of resin composites to enhance the mechanical properties of them is the application of nanotechnology to resin composites. Nanotechnology is defined as the development of new structures and materials in the range of approximately 0.1-100 nanometers that lead to different chemical or physical characteristics. In nanohybrid composites, an increased filler load may be obtained due to the reduced dimensions of the particles and a greater size distribution of fillers and lead to decreased polymerization shrinkage and promotion of mechanical properties of composites such as tensile and compressive strength, and fracture resistance. These characteristics appear to be equivalent or sometimes higher than those of

universal hybrid or microfilled composites [5-7]. Calcium enriched mixture (CEM) was introduced [8] as an endodontic material that has the same clinical applications but different chemical composition from mineral trioxide aggregate (MTA). This cement like MTA releases calcium hydroxide during and after setting [9, 10]. CEM has similar antibacterial features as calcium hydroxide, but better than MTA [11]. Its sealing ability, pH, and cytotoxic effects are similar to MTA; however, it has increased flow, decreased setting time, and film thickness. This cement has excellent biocompatibility and tremendous capacity to induce hard tissue formation in vital pulp therapies [12, 13]. CEM has shown acceptable results in pulpotomy of permanent teeth, apexogenesis, management of furcal perforation, and internal and external root resorption [14-16].

CEM cement appears to be a preferable alternative to MTA because of easier manipulation and increased thickness of the dentin bridge with CEM cement compared to MTA [13, 17]. The shear bond strength of vital pulp capping materials to restorative materials has been a critical issue in recent years. The bond strength of vital pulp capping materials and composite resins, affect the quality of filling and success of restoration. On the other hand, the proper bonding of composite resins to vital pulp capping biomaterials result in the adhesive joint to spread the stress relatively evenly over the entire surface of the bond [18].

The present study is designed to compare the shear bond strength of silorane and nanohybrid composite resin to CEM cement in two different time periods. We intend to determine whether the time of applying the composite resin on the CEM cement can affect shear bond strength of these materials.

Materials and Methods

Table 1 lists the compositions of the materials used in this study. We prepared 60 CEM cement (Yektazist Dandan, Iran, Tehran) specimens by using cylindrical acrylic blocks. Each block had a central hole that measured 4 mm in diameter and 2 mm in depth. The CEM cement was mixed according to the manufacturer's instructions, poured into the holes in the acrylic blocks, and

Table 1: Test materials, manufacturers, and composition

Name	Manufacture	Type	Composition
Silorane adhesive system primer	3M, ESP, ST. PAUL, MN, USA	Two-step self-etch acid primer	Phosphorylate methacrylates, vitrebond copolymer, bis-GMA, HEMA, water/ethanol solvent, silane treated silica fillers
Silorane adhesive system bond	3M, ESP, St. Paul, MN, USA	Two-step self-etch acid primer	Phosphorylate methacrylates, hydrophobic dimethacrylate, TEGDMA, silane treated silica fillers
Filtek [®] P90	3M, ESP, St. Paul, MN, USA	Silorane-based composite resin	Monomers:3,4-epoxycyclohexyl-ethyl-cyclo-poly-methylsiloxane (5%-15% w/w), bis-3,4-epoxycyclohexyl-ethyl-phenyl-methylsilane (5%-15% w/w); Fillers: SiO ₂ , YtF ₃ (55% v, 76% w)
Etchant 37%	Ivoclar Vivadent, Germany	-	H ₃ PO ₄ 37%
Adper single bond II	3M, ESP, St. Paul, MN, USA	Two-step self-etch acid primer	Bis-GMA, HEMA, dimethacrylated, polyalkenoic acid copolymer, water/ethanol solvent
Filtek [®] Z350	3M, ESP, St. Paul, MN, USA	Methacrylate-based composite resin	Monomers: Bis-GMA, UDMA, TEGDMA, PEGDMA, bis-EMA
CEM cement	Yektazist Dandan, Iran	Pulp capping material	51.75% w calcium oxide (CaO), 9.53% w sulfur trioxide (SO ₃), 8.49% w phosphorous pentoxide (P ₂ O ₅), 6.32% w silicon dioxide (SiO ₂), aluminum trioxide (Al ₂ O ₃), sodium oxide (Na ₂ O), magnesium oxide (MgO), chloride (Cl)

Abbreviations; Bis-GMA: Bisphenol-a-glycidyl dimethacrylate, HEMA: Hydroxyethyl methacrylate, TEGDMA: Tetraethyleneglycol dimethacrylate, UDMA: Urethane dimethacrylate, PEGDMA: Polyethylene glycol dimethacrylate, bis-EMA: Ethoxylated bisphenol-a-glycol dimethacrylate

covered with a moist cotton pellet and temporary filling material (Cavit; ESPE America, Norristown, PA, USA).

We stored 30 specimens for 3 days and the remaining 30 specimens were stored for 7 days at 37°C and 100% humidity. After removal of the temporary material and moist cotton, we did not rinse or polish the CEM cement surface. The specimens in each group were further divided into 2 subgroups of 15 specimens each. Preparation of the specimens was based on the type of the employed adhesive system and the time interval as follows:

Group 1 (3 day): CEM cement + acid etching + Adper Single Bond + Composite Z350

Group 2 (7 day): CEM cement + acid etching + Adper Single Bond + Composite Z350

Group 3 (3 day): CEM cement + Silorane adhesive system + Composite P90

Group 4 (7 day): CEM cement + Silorane adhesive

system + Composite P90

After removing the temporary material and moist cotton, nanohybrid (Z350) and silorane (P90) composite resins were applied on the CEM cement surface by packing the materials into a split cylindrical metal mold with an internal diameter of 3 mm and a height of 2 mm. The specimens were cured with an LED curing system (Demi Plus, Kerr, Switzerland) for 20 seconds within the split mold with an 800 mW/cm² light intensity and 20 seconds after split mold removal to ensure complete setting of the whole composite. After this process, specimens were stored at 37°C and 100% humidity for 24 hours in an incubator (Binder 7200 Tuttlingen, Germany) to encourage setting. All samples were prepared by the same operator. The specimens were mounted in a universal testing machine (Zwick/Roell Z020, Stuttgart, Germany) and we evaluated the shear force. A crosshead

speed of 0.5 mm/min was applied to each specimen by a knife-edge blade until the bond between the CEM cement and composite resins failed. The values were calculated in newtons and converted into megapascals (MPa). Next, the samples were evaluated under a stereomicroscope (Motic K-500L, Motic Inc. Ltd., Hong Kong) under 40x magnification. An expert observed confirmed and categorized the failure modes as either adhesive (between two materials), cohesive (inside one of the materials), or mixed (both adhesive and cohesive).

Statistical analysis

The means and standard deviations were calculated. The mean bond strengths of the groups were compared by two-way analysis of variance (ANOVA). Two-way ANOVA was employed to assess the effect of composite resin type at different time intervals. Due to the significant interaction effect, we performed subgroup analysis by the student's t-test. $P < 0.05$ was considered statistically significant.

Results

Table 2 and Figure 1 demonstrate the mean shear bond strength values and standard deviation of the

Table 2: Descriptive statistics of the groups and comparison of shear bond strength values (MPa; n=15)

Groups	Code	Mean±SD	Significance
1	A	2.12±0.66	A,C (P=0.004)
2	B	1.92±0.39	B,D (P=0.011)
3	C	3.12±0.75	C,D (P=0.0002), C,A (P=0.004)
4	D	1.46±0.42	D,C (P=0.0002), D,B (P=0.011)

two composite systems at the two time intervals. Two-way ANOVA and the student's t-test showed a significant interaction effect between composite type and time interval ($P < 0.001$). Thus, the effect of time on shear bond strength depended on the type of composite resins. At day 3, shear bond strength of P90 (3.12±0.75) was significantly higher than Z350 (2.12±0.66; $P = 0.001$). However, at day 7, Z350 (1.92±0.39) had significantly higher shear bond strength compared to P90 (1.46±0.42; $P = 0.004$). Different time intervals affected the shear bond strength of P90 composite resins, as the mean shear bond strength of composite P90 at day 3 (3.12±0.75) was significantly higher than the mean

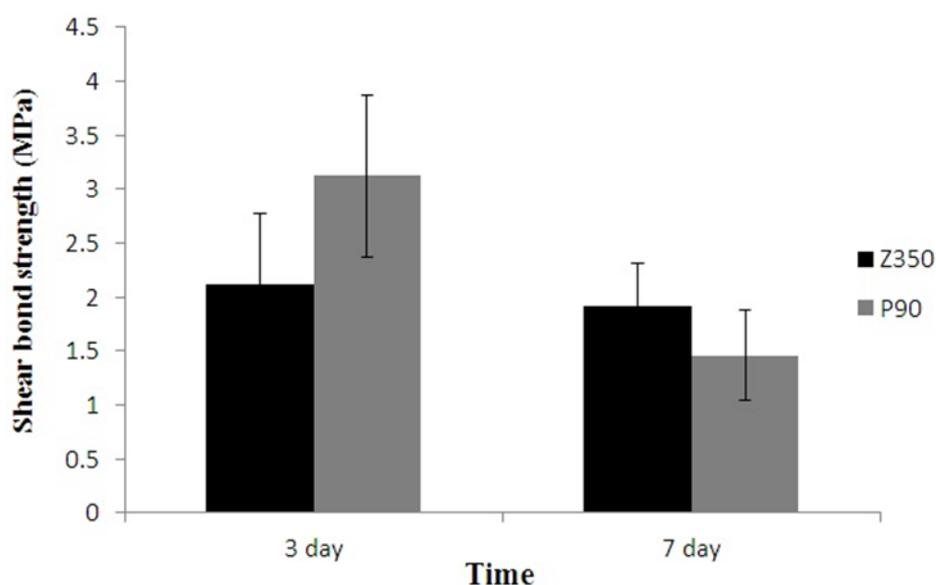


Figure 1: Mean shear bond strength (MPa) of the P90 and Z350 groups at different time periods

shear bond strength at day 7 (1.46 ± 0.42 ; $P < 0.001$). On the other hand, different time intervals did not show a significant change on the shear bond strength of Z350 ($P = 0.341$). Evaluation with stereomicroscope showed cohesive failure in 82% and adhesive failure in 8% of the samples. Mixed failures were seen in 10% of the specimens (Table 3). Differences between cohesive and adhesive failure modes were significant. Differences between cohesive failures and mixed failures were also significant. Data were analyzed by the student's t-test ($P < 0.05$).

Table 3: Frequency of specimen failure modality

Groups	Cohesive	Adhesive	Mixed
1	9	0	2
2	12	2	1
3	15	1	2
4	13	2	1

Discussion

One of the most important factors necessary for success of vital pulp therapy is insertion of a perfect restoration as soon as possible after endodontic treatment in order to make and maintain an acceptable seal. Composite restoration is recommended as the result of lower forces applied onto the pulp capping biomaterial during placement of restoration. Therefore the bond strength between pulp capping biomaterials and composite resins has a crucial role in quality of fillings and treatment outcome [19]. One of the most commonly used methods for evaluation the bond strength of dental materials to composite resins is the shear bond strength test [19]. Therefore, we have used the shear bond test to evaluate the adhesive joint between CEM cement biomaterial and two types of adhesive systems. The bond strength of pulp capping biomaterials such as CEM cement to composite resin depends on the physical and chemical characteristics [20, 21]. CEM cement is a hydrophilic biomaterial which sets in the presence of moisture. The main ingredients of CEM include calcium oxide, sulfur trioxide, silica, and phosphorous [10]. CEM

is able to precipitate hydroxyapatite even when stored in normal saline. Because CEM cement does not have resin components, the bonding mechanism of it to composites is probably micromechanical and the result of penetration and interlocking of the composite adhesive system into the surface irregularities and pores [22, 23]. The polymerization reaction mechanism of composite resins is associated with polymerization shrinkage. The available composite resins have polymerization shrinkage in the range of 2.9% to 7.1%, depending on the test. Polymerization shrinkage can result in shrinkage stresses up to 7 MPa. Composite resins that do not undergo polymerization shrinkage are not available in the market. Research is underway for producing composite resins with low or no polymerization shrinkage. Presently, silorane system composites have been introduced as a new dental composite that have polymerization shrinkage of less than 1%, which may influence its bond strength [24]. Adhesion in self-etch silorane adhesive is created by acidic monomers that etch dental substrates and thus create a retention pattern for micromechanical interlocking of the cured adhesive with the tooth. They provide chemical bonding to the calcium-containing dental materials. The majority of current self-etch adhesives contain phosphorylated methacrylates as acidic monomers. Some contain carboxylic acid functionalized monomers or the combination of both. Silorane System Adhesive primer contains phosphorylated methacrylates, as well as the vitrebond copolymer with its carboxylic acid functionality used in some types of resin-modified glass ionomers and adhesives for adhesion to dentin and enamel. Comonomers, like bisphenol-a-glycidyl dimethacrylate (bis-GMA) and hydroxyethyl methacrylate (HEMA), a solvent system that consists of water and ethanol for wetting and penetrating the dental substrates, and a photoinitiator system based on camphorquinone for thorough and fast curing. A silane treated silica filler with a primary particle size of approximately 7 nm has been added to improve the mechanical strength and film-forming properties of the Silorane System Adhesive Self-Etch Primer. This filler is very finely dispersed in order to prevent settling [25]. This study showed that the shear bond strength of the P90 low shrinkage adhesive system was significantly higher than the Z350 nanohybrid

composite at day 3. This finding might be due to the higher amount of the acidic primers that contain acetone or alcohol, which might affect the characteristics of the CEM cement. These acidic primers could react with the phosphorus component of CEM cement; however, after a number of days, these acidic monomers might undergo hydrolyzed degradation. The self-etch and hydrophilic nature of the silorane adhesive system is compatible with the hydrophilic nature of CEM cement. After a number of days, hydrolyzed degradation of the self-etch adhesive layer can occur and may lead to a decrease in shear bond strength. On the other hand, because of less polymerization shrinkage (about 1%) of the P90 composite, we have lesser gap formation between the composite and CEM cement that lead to increase shear bond strength. With a pH of approximately 2.7, the silorane system adhesive primer provides rather mild etching and produces a nanoetching pattern, as well as chemical bonding to the calcium and phosphate of CEM cement [26]. It can create mechanical interlocking between the bonding agent and porosity caused by mild etching of the CEM cement surface. The acid etching process in total etch adhesive systems may have some effects on setting time of the pulp capping biomaterials. This delay in setting of CEM cement may lead to lower bond strength of total etch adhesive systems at day 3. It seems that in phosphate-buffered solution, hydroxyapatite crystals are precipitated over CEM biomaterials. Although the particle sizes of CEM biomaterials differ, the pH and setting/working times are similar [27]. Previous studies with scanning electron microscope (SEM) results show that the surface gel-like amorphous structure and needle-like crystals are removed during acid etching. The selective removal of matrix from the periphery of crystals results in a spongy appearance, which can produce a good surface for bonding [28]. A few studies investigated the bonding strength of CEM cement biomaterial to silorane-based composite resins. Oskoe *et al* evaluated the bond strength of CEM cement and MTA cement with Z250 methacrylate base composite with and without acid etching. The results showed no significant differences in bond strength values of CEM cement and MTA to composite resin. They concluded that surface etching of MTA and CEM biomaterials

were not mandatory prior to composite restoration using total-etch adhesive resin during vital pulp therapy [20].

It has been reported by Boushell that the silorane composite resin had significantly lower bond strength to resin modified glass ionomer (RMGI) compared to methacrylate composite resin. However, in the current study, silorane composite resin showed higher bond strength. The differences observed in this study might be related to using a silorane adhesive system with CEM cement, not with RMGI or dentin surface [29]. In accordance with our study Kasraie *et al.* showed that application of the self-etch system lead to a more significant increase in micro shear bond strength between the RMGI and composite resin compared to the total etch system [30]. Krifka *et al.* also showed that in some adhesive systems the bond strength was influenced by time [31]. The technique of a CEM cement sample preparation can affect the bond strength results. In this study, the surface of CEM cement was not rinsed or polished, which might lead to lower bond strength compared with the previous studies that investigated other cements. We did not polish the CEM cement surface, because obtaining a smooth, glazed cement surface under composite resin could not be reproduced in clinical conditions. In contrast with the current study, two studies reported no significant differences in shear bond strength between total etch and self-etch adhesive systems [31]. These findings might be due to the differences in the type of adhesives and perhaps the method for their application. One of those studies used the adhesive systems, Protect bond and SE Bond, both of which have different pH and different compositions from the silorane self-etch adhesive system. On the other hand, because of less polymerization shrinkage (approximately 1%) of the P90 composite, the gap formation decreased at first and lead to increased shear bond strength. However, in the nanohybrid Z350 composite that has a total etch hydrophobic adhesive nature, we observed more polymerization shrinkage and gaps on day 3. This perhaps led to lower shear bond strength; however, after 7 days, the moisture absorption by the total etch adhesive system might lead to decreased gaps and increased shear bond strength [4, 32, 33]. The findings of this study have shown lower bond strength between

CEM cement and composite resins compared to other studies that evaluated other dental cements. In order to fulfill a favorable restorative procedure when two different materials are used, an appropriate bond between these materials should be considered. Generally, when fracture occurs inside each material (cohesive failure) the bond is considered acceptable, rather than in the interface (adhesive failure) [34]. Nearly all failures in the present study were the cohesive type that occurred inside the materials, which might show the low cohesive strength of the materials compared to their bond strength. In a study by Oskoe *et al*, there was no significant difference between shear bond strength of MTA and CEM cement to adhesive resin cylinders using Single Bond. The researchers evaluated the shear bond strength of MTA and CEM cement to composite resin with and without the use of acid etch [20]. Another study also showed that the failure modes between CEM cement and dentin were mostly the cohesive type, which agreed with the results of the present study [35]. More studies should be conducted to investigate other aspects of bond quality and the influence of time on bond strength of pulp capping biomaterials to restorative dental materials.

Conclusions

Within the limits of the present study, we observed the best results of P90 silorane based composite filling after vital pulp therapy with CEM cement biomaterial at day 3, whereas the Z350 nanohybrid composite showed better results at day 7.

Acknowledgments

The authors would like to thank the Vice Chancellor of Shiraz University of Medical Sciences for supporting this research (Grant# 8218), which is based on the thesis by Dr. Masoud Esfandiari.

Conflict of Interest: None declared.

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