Comparison of shear bond strength of light cure mineral trioxide aggregate and light cure calcium hydroxide with nanofilled composite: A stereomicroscopic and scanning electron microscope analysis

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ABSTRACT

Objective: The quest for ideal pulp capping materials has given rise to the development of newer materials such as light cure mineral trioxide aggregate (MTA). The bond strength of the pulp capping materials with overlying restoration is one among the several factors that are critical for the success of vital pulp therapy. Hence, we conducted this study to evaluate and compare the shear bond strength (SBS) of light cure MTA and light cure calcium hydroxide with nanofilled composite. Materials and Methods: Thirty acrylic blocks each with a central hole were prepared to uniform dimensions and randomly distributed into two equal groups. In Group I, light cure MTA, and in Group II, light cure calcium hydroxide was used as pulp capping materials. After the application of adhesive system, nanofilled composites were applied onto the pulp capping material using a cylindrical plastic matrix. The SBS was tested on a universal testing machine (Instron 3366, UK) at a crosshead speed of 0.5 mm/min. The samples were examined under stereomicroscope and scanning electron microscope to analyze different modes of failure. Results: The results were statistically analyzed using independent sample t-test. Light cure MTA attained the mean SBS of 6.54 MPa and light cure calcium hydroxide attained the mean SBS of 6.56 MPa. There was no significant difference statistically in SBS of both the materials (P < 0.05). The modes of failure were predominantly mixed failure followed by cohesive failure within the restorative material in both Group I and II. Conclusion: The results of the study suggest that the SBS of light cure MTA and light cure calcium hydroxide is comparable. The modes of failure analyzed in both light cure MTA and light cure calcium hydroxide are not significantly different statistically. Hence, both materials can be successfully used as pulp capping material with nanofilled composite.

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KEYWORDS: Dental pulp, nanofilled composites, pulp capping, shear bond strength

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Introduction

Vital pulp therapy (VPT) is a biologic and conservative treatment modality to preserve the vitality and health of the dental pulp following carious exposure or traumatic injury.\(^1\)\(^-\)\(^6\) It includes two therapeutic approaches namely indirect pulp therapy for deep dentinal caries and direct pulp capping or pulpotomy in cases of pulp exposure.\(^5\)

The success of VPT depends on the therapeutic property of the pulp capping material used and its compatibility to underlying pulp and overlying restorative material.

Calcium-based material has dominated the genera of pulp capping material due to their favorable biocompatibility, antibacterial property, and its bioactivity in terms of formation of hard tissue barrier.\(^4\)\(^-\)\(^7\) The self-curable preparations were highly soluble and also lacked inherent adhesive properties which led to failure of the pulp therapy.\(^8\)\(^-\)\(^10\) However the advent of light cure calcium based pulp capping materials revolutionised the scenario of vital pulp therapy. The light curable calcium-based materials are likely to offer better adhesive property along with therapeutic benefits of calcium in pulpal healing and hence are expected to amplify the success of VPT.

The adhesive compatibility in terms of shear bond strength (SBS) of pulp capping material to overlying restoration is another critical factor in determining the success of VPT. Nanofilled composites offer a favorable choice of material to produce a monolithic reconstruction and a microleakage-free restoration. Since nanofilled composites are composed of nanosized fillers, they are known to exhibit better mechanical and physical properties than conventional composite restorations.\(^11\) Hence, we conducted this \textit{in vitro} study to evaluate and compare the SBS of two different calcium-enriched mixtures of pulp capping materials, namely light cure mineral trioxide aggregate (MTA) and light cure calcium hydroxide with nanofilled composites. A null hypothesis stating that “there is no difference in the SBS of light cure MTA and light cure calcium hydroxide with nanofilled composites” was adopted. The objective of the study was to determine a compatible pulp capping and restorative material to enhance the success rate of VPT.

Materials and Methods

This was an experimental type of \textit{in vitro} study with a sample size of 30 acrylic blocks. The sample size was estimated at 95% confidence level with a power of 80 based on previous studies. Of the 30 acrylic blocks, 15 were filled with light cure MTA and 15 with light cure calcium hydroxide, which served as the samples in Group I and II, respectively.

Specimen preparation

\textbf{Preparation of acrylic blocks}

Acrylic blocks with dimensions 50 mm in height, 20 mm in width, and 15 mm in thickness were prepared using cold cure methyl methacrylate resin and polished with 220, 320, 400, and 600 grit carbide polishing paper. Following this, a central hole of 4 mm in deep and 5 mm in diameter was drilled into the polished surface of the acrylic block,\(^12\) with grooves added for additional retention. An impression of this block was taken using silicone elastomeric impression material, which was used as a mold for fabrication of acrylic blocks to maintain uniformity between all the blocks. Thirty such acrylic blocks were then prepared.

The acrylic blocks were then randomly distributed into two groups of 15 blocks each.

- **Group I** \((n = 15)\) – 15 blocks were filled with light cure MTA
- **Group II** \((n = 15)\) – 15 blocks were filled with light cure calcium hydroxide.

\textbf{Preparation of samples with light cure mineral trioxide aggregate as base material – Group I}

Light cure MTA (TheraCal LCT\(^{TM}\)) was injected into the central hole of the acrylic resin block. The material was covered with a glass microscopic slide to produce a smooth surface and light cured for 20 s. The surface was etched with 37% phosphoric acid etchant (Eco-Etch\(^{TM}\), Vivadent, Lichtenstein) for 15 s. A single layer of bonding agent was applied to the surface and light cured for 20 seconds with a blue light-emitting diode curing unit. Following this, composite resin was placed.

\textbf{Preparation of samples with light cure calcium hydroxide as the base material – Group II}

Light cure calcium hydroxide was injected into the central hole of the acrylic resin block. The material was covered by a glass microscopic slide and then cured for 20 s. Acid etching and bonding agent was applied as for samples prepared in Group I followed by the placement of composite resin over the surface.

\textbf{Placement of restorative material}

The composite material (Filtek Z350 XT, 3M ESPE, St. Paul, MN, USA) was applied into a cylindrical-shaped plastic matrix with an internal diameter of 4 mm and height of 2 mm. This was followed by light curing with an intensity of 1200 mV/cm\(^2\) for 20 s. The prepared specimens were stored at 37°C in 100% humidity for 24 h for setting.\(^12\)

\textbf{Shear bond strength test}

SBS testing was carried out by placing the prepared specimens in the lower platform of the universal testing machine (Instron 3366, UK) and was held firmly in position using a specially designed jig for this purpose as shown in Figure 1. Subsequently, a chisel-shaped debonding blade was attached to the crosshead of the
universal testing machine. The load was applied at the interface between the materials bonded at a crosshead speed of 0.5 mm/min until the debonding occurred. The maximum load observed during the test divided by the bond area was considered as SBS and was expressed in MPa. The data obtained were subjected to statistical analysis.

Evaluation of fracture patterns
The fractured test specimens were examined under a stereomicroscope at ×25 and the fractures were classified as follows: (1) cohesive failure - When the failure occurred within restorative material/pulp capping material; (2) adhesive failure - When the failure occurred at the interface between light cure MTA/light cure calcium hydroxide and restorative material; and (3) mixed failure - When a combination of adhesive fracture and the cohesive fracture was evident.

Scanning electron microscopy
After examining the specimens under a stereomicroscope, the three representative samples in each group were reduced in size. The samples were then attached to a base that had six slots with the help of thin double tape. The base was then placed into scanning electron microscope and viewed at ×500. The images were observed to evaluate the features of the interface between the two bonding surfaces at the site of failure.

Statistical analysis
The data obtained after evaluation of test parameters were subjected to statistical analysis. All the analysis was done using SPSS version 18 (SPSS Inc., Chicago, USA).

Results
Shear bond strength
Group I (light cure mineral trioxide aggregate-nanofilled composite)
SBS between light cure MTA and nanofilled composite in the samples of Group I measured the highest value of 10.06 MPa, with a mean value of 6.5440 and standard deviation of 2.64366.

Group II (light cure calcium hydroxide-nanofilled composite)
SBS between light cure calcium hydroxide and nanofilled composite in the samples of Group II measured the highest value of 9.62 MPa, with a mean value of 6.5600 and standard deviation of 1.96866.

Intergroup comparison of the SBS between Group I and II was statistically analyzed using SPSS version 18. The P < 0.05 was considered statistically significant. Comparison of mean values was done using independent sample t-test as shown in Table 1. The normality of the distribution was done using Levene’s test as shown in Table 2.

Fracture analysis
The samples in Group I and Group II were examined under stereomicroscope at ×25 for adhesive and cohesive failure modes.

In Group I, one sample showed adhesive failure, six samples showed cohesive failure of which five were within the restorative material and one was within the capping material, while eight samples showed combination of adhesive and cohesive failures. A representative sample from the Group I showing cohesive failure within the light curable MTA wherein failure has occurred within the capping material is

| Table 1: Intergroup comparison of shear bond strength between Group I (TLC-NCR) and Group II (CLC-NCR) using independent sample t-test |
|---------------------------------------------------------------|
| **Groups** | **n** | **Mean** | **SD** |
| MCS | TLC | 15 | 6.5440 | 2.64366 |
| | CLC | 15 | 6.5600 | 1.96866 |

SD=Standard deviation; TLC=Light cure MTA; CLC=Light cure Calcium hydroxide; NCR=Nano filled composite; MCS=Mean compressive strength

Figure 1: Specimen mounted on Instron machine for shear bond testing

Figure 2: Stereomicroscopic image of representative sample from Group I (TLC-NCR) showing cohesive failure within TheraCal LC™
shown in Figure 2. A representative sample from the Group I showing cohesive failure within composite wherein the failure has occurred within the restorative material is shown in Figure 3. A representative sample from the Group I showing mixed failure wherein combination of both adhesive and cohesive failure has occurred is shown in Figure 4. A representative sample from Group I showing adhesive failure wherein failure has occurred at the site of light curable MTA is shown in Figure 5.

In Group II, one sample showed adhesive failure, five samples showed cohesive failure of which two were within the capping material and three were within the restorative material, while nine samples showed a combination of both adhesive and cohesive failures. A representative sample from Group II showing mixed failure wherein the combination of both adhesive and cohesive failure has occurred is shown in Figure 6. A representative sample from Group II showing adhesive failure wherein failure has occurred at the site of light cure calcium hydroxide is shown in Figure 7. A representative sample showing cohesive failure within light cure calcium hydroxide in Group II wherein failure has occurred within the capping material is shown in Figure 8. A representative sample

![Figure 3: Stereomicroscopic image of representative sample from Group I (TLC-NCR) showing cohesive failure within composite](image)

![Figure 4: Stereomicroscopic image of the representative sample from Group I (TLC-NCR) showing a combination of adhesive and cohesive failure](image)

![Figure 5: Stereomicroscopic image of representative sample from Group I (TLC-NCR) showing adhesive failure](image)

![Figure 6: Stereomicroscopic image of the representative sample from Group II (CLC-NCR) showing a combination of adhesive and cohesive failure](image)

| MCS | Levene’s test for equality of variances | t-test for equality of means |
|-----|----------------------------------------|-------------------------------|
|     | $F$ | Significant | $t$ | df | Significant (two-tailed) | 95% CI of the difference |
| Equal variances assumed | 0.586 | 0.450 | 0.019 | 28 | 0.985 | $-1.72732$ | $1.79932$ |

CI=Confidence interval; TLC=Light cure MTA; CLC=Light cure Calcium hydroxide; NCR=Nano filled composite; MCS=Mean compressive strength
showing cohesive failure within composite in Group II wherein failure has occurred within the restorative material is shown in Figure 9.

**Scanning electron microscopic observations**

In Group I, the scanning electron microscopic images of cohesive failure occurred within the capping material are depicted in Figure 10. The surface showed fracture lines seen within light curable MTA. The scanning electron microscopic image of a combination of cohesive and adhesive failure is depicted in Figure 11. The surface showed fracture lines at the interface as well as within light curable MTA.

In Group II, the scanning electron microscopic images of cohesive failure occurred within the composite are depicted in Figure 12. The image showed smooth surface indicating failure has occurred within the composite. The scanning electron microscopic image of a combination of cohesive and adhesive failure is depicted in Figure 13. The surface showed fracture lines at the interface as well as within the light cure calcium hydroxide.

**Discussion**

Several studies have investigated the sealing ability, cytotoxicity, calcium-releasing ability, dentin bridge formation, and other physical and chemical properties of light cure MTA and light cure calcium hydroxide.[9,10,13-16] The bond between light cure MTA and light cure calcium hydroxide with nanofilled composite or overlying restoration is of pivotal importance for the success of VPT.

Therefore, the present study was planned with the aim of evaluating and comparing the SBS of two pulp capping materials to nanofilled composite. In this study, the macro-SBS of light cure MTA and light cure calcium hydroxide with nanocomposite was evaluated using universal testing machine. According to Salz
macroshear bond test is the most commonly used technique to test bonding and is used in 26% of scientific papers reporting on bond strength.[18] The popularity of this testing method is a result of its ease and speed, as well as the lack of specimen processing requirements as compared to other approaches. This method was thus chosen for testing the bond strength, as our goal was to find the bond strengths of two pulp capping materials with the overlying restorative composite resin.

The results of SBS performed in our study showed the mean SBS of 6.54 and 6.56 MPa in Group I (light cure MTA-nanofilled composite) and Group II (light cure calcium hydroxide-nanofilled composite), respectively. The mean SBS between light cure MTA-nanofilled composite and light cure calcium hydroxide-nanofilled composite showed no statistically significant difference.

In the study by Cantekin, they evaluated the bond strength of methacrylate-based (MB) composites, silorane-based (SB) composites, and glass-ionomer cement (GIC) in comparison to light cure MTA and compared those findings with the reference pulp capping material (MTA). They concluded that the mean SBS of MB composite with light cure MTA was 19.3 MPa, SB composite with light cure MTA (TheraCal) showed low SBS of 3.6 MPa, and GIC with light cure MTA showed an SBS of 3.4 MPa.[12]

Alzraikat et al. compared the SBS of light cure MTA to resin composite with that of MTA using two bonding systems (i.e., etch and rinse system and 1-step self-etch adhesive system). In this study, light cure MTA showed SBS of 12.8 and 14.78 MPa for etch and rinse system and 1-step self-etch adhesive, respectively.[2]

However, the results of our study demonstrated an SBS value of 6.54 MPa with light cure MTA and 6.56 MPa with light cure calcium hydroxide with overlying layer of nanofilled composite. This difference in SBS value obtained in our study may be attributed to the different composite material and different etching and bonding protocols used.[19] The type of composite material used has been reported to have a significant influence on bond strength.[20]

The difference in SBS of light cure MTA with etch and rinse system may be due to surface area available for bonding. As in the current study, the surface area available was 5 mm in diameter compared to the study conducted by Alzraikat et al. and Cantekin, wherein the surfaces available were 3.5 mm and 2 mm diameter, respectively.[21] It is well established that larger surface area for bonding will produce lower SBS values because of increased probability of the presence of critical-sized defects.[22]

The other factor that can be attributed to the lower bond strength achieved in our study may be due
to the adhesive system used. Application of a silane coupling agent has been suggested to improve the bonding between the tooth and restorative materials. The bifunctional silane molecule bonds chemically to silica-containing materials and have methacrylate functionality that allows chemical union with the resinous substrate. Silanes also act as adhesion promoters by enhancing the wetting ability of the adhesive system.[22] This gives additional chemical bonding with calcium releasing bioactive liners. Silanes were absent in the adhesive system used in our study. Therefore, a lower SBS value may have been obtained.

Since there are no published data to compare the bond strength achieved by light cure MTA and light cure calcium hydroxide to overlying nanofilled composite, a direct comparison for results of SBS between the two as obtained in our study cannot be correlated to other studies.

When two different materials are used, the success of restoration depends on appropriate bond between the two materials. The bond between pulp capping agent and restoration fails when the load exceeds the SBS between pulp capping agent and restorative material. In a situation where the bond strength between the two materials exceeds the strength of individual materials, failure often occurs cohesively in either of the materials. In contrast, adhesive failure at the bonding interface will occur when the bond strength is less than the strength of the materials. Therefore, cohesive failures are more acceptable than adhesive failures.[23]

In our study, modes of failure were analyzed using stereomicroscope and scanning electron microscopy. When interpreting the failure mode, there is neither clear agreement in the literature regarding their classification nor have these failure modes been analyzed based on the same microscopic analytical tools. The modes of failure are often only evaluated with low power microscope magnification, which adds to the errors of interpretation of the materials involved at the fractured surface and to the distinction of failure modes. Hence, both stereomicroscopy and scanning electron microscopy were used to evaluate the type of failure in our study. Large cohesive failures within resin can be well evaluated with a stereomicroscope at low magnification. However, the decision on the mode of failure for the adhesive interface or mixed failures can be evaluated better using a scanning electron microscope at higher magnification.[24]

In our study, analysis of modes of failure showed a greater number of samples exhibiting mixed and cohesive failures in both the groups (light cure MTA-nanofilled composite and light cure calcium hydroxide and nanofilled composite). Mixed failure indicates the bond between the materials is not uniform across the samples. Several studies have concluded that better bond between two materials is achieved when a higher percentage of cohesive and mixed failures were observed.[25,26] In our study, the specimen showing higher bond strength within the groups correlated to cohesive and mixed failure modes when observed under stereomicroscope. The results on failure modes in our study are in agreement to a study by Alzraikat et al.[2] Since only one sample in each group showed adhesive failure, it suggests that light cure MTA and light cure calcium hydroxide bond well with nanocomposite. This may be attributed to resin content in light cure MTA and light cure calcium hydroxide, which provides a bond with nanofilled composite. Moreover, the hydrophilic resin monomer in light cure MTA makes it an excellent adhesion promoter enhancing bond strength.[2]

Based on the results of the current study, the null hypothesis as stated as “There is no difference in SBS between light cure MTA and light cure calcium hydroxide with nanofilled composite” was accepted.

Since an in vitro study cannot simulate in vivo conditions, extrapolation of the results of the present study needs to be carefully done. In in vivo, the type of shear stress and the load exerted on the tooth and restoration are different compared to a standardized load applied in in vitro studies. Long-term clinical trials are required to assess the efficacy of light cure MTA and light cure calcium hydroxide as pulp capping agents. Further research is indicated with greater sample size to conclude the results achieved in this study.

Conclusion

As per the observations of our study, we found no statistical difference in SBS between light cure MTA with nanofilled composite and light cure calcium hydroxide with nanofilled composite. Therefore, we can conclude that both light cure MTA and light cure calcium hydroxide can be successfully used as pulp capping agents with nanocomposite.

The analysis of failure mode showed a greater number of samples in both the groups exhibited mixed failures and cohesive failures. The adhesive failures were not reported in both the groups, which indicate a strong adhesive bond.

For the durability and longevity of the final restoration, it is important that adequate bonding is achieved between the composite resin and the pulp capping agent. The simple application and high bond strength capacity of light cure MTA and light cure calcium hydroxide to nanofilled composite support its use and consideration as the material of choice in VPT.

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Conflicts of interest
There are no conflicts of interest.

References
1. Akhlaghi N, Khademi A. Outcomes of vital pulp therapy in permanent teeth with different medicaments based on review of the literature. Dent Res J (Isfahan) 2015;12:406-17.
2. Alzraikat H, Taha NA, Qarsawi D, Burrow MF. Shear bond strength of a novel light cured calcium silicate based-cement to resin composite using different adhesive systems. Dent Mater J 2016;35:881-7.
3. Fuks AB. Vital pulp therapy with new materials for primary teeth: New directions and treatment perspectives. J Endod 2008;34:S18-24.
4. Savas S, Botsali MS, Kucukyilmaz E, Sari T. Evaluation of temperature changes in the pulp chamber during polymerization of light-cured pulp-capping materials by using a valo led light curing unit at different curing distances. Dent Mater J 2014;33:764-9.
5. Arandi NZ. Calcium hydroxide liners: A literature review. Clin Cosmet Investig Dent 2017;9:67-72.
6. Estrela C, Holland R. Calcium hydroxide: Study based on scientific evidences. J Appl Oral Sci 2003;11:269-82.
7. Subramaniam P, Konde S, Prashanth P. Evaluation of temperature variations in calcium hydroxide liners. J Indian Soc Pedod Prev Dent 2006;24:144-5.
8. Hilton TJ. Keys to clinical success with pulp capping: A review of the literature. Oper Dent 2009;34:615-25.
9. Alhadiainy HA, Himel VT. Comparative study of the sealing ability of light-cured versus chemically cured materials placed into furcation perforations. Oral Surg Oral Med Oral Pathol 1993;76:338-42.
10. Yalcin M, Barutcigil C, Sisman R, Yavuz T, Orucoglu H. Evaluation of sealing ability of pulp capping agents against leakage on direct pulp capping with a computerized fluid filtration meter. J Restorative Dent 2014;2:46-50.
11. Mikhail SS, Schrickler SR, Azer SS, Brantley WA, Johnston WM. Optical characteristics of contemporary dental composite resin materials. J Dent 2013;41:771-8.
12. Cantekin K. Bond strength of different restorative materials to light-curable mineral trioxide aggregate. J Clin Pediatr Dent 2015;39:143-8.