

BONDING ABILITY OF DIFFERENT LINERS TO BULK-FILL RESIN COMPOSITE USING SILANE-CONTAINING ADHESIVE

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ABSTRACT

Aims: To compare and evaluate the bonding ability of bulk-fill resin composite (RC) to four different liners: Biodentine (BD), TheraCal (TLC) - a novel resin-modified calcium silicate cement, resin-modified glass ionomer cement (RMGIC) and conventional glass ionomer cement (GIC) using a universal silane-containing adhesive and characterizing their failure modes.

Materials and Methods: Forty extracted intact human premolars with occlusal cavity (4-mm diameter and 2-mm height) were mounted in acrylic blocks and divided into four groups of (n=10 samples) each based on the liner used as group I; (BD), group II; (TLC), group III; (RMGIC) and Group IV; (GIC). Bulk-fill composite buildup of 3 mm diameter and 5 mm height was then bonded to each sample using universal adhesive. Shear bond strength (SBS) analysis was performed using materials testing machine at a cross-head speed of 0.5 mm/min.

Statistical Analysis: Statistical analysis was performed with one-way analysis of variance (ANOVA) and Tukey's post hoc test for numerical data while chi square test for categorical one.

Results: One-way analysis with ANOVA revealed significant difference in bond strength values between the different groups ($p < 0.001$). The observed modes of failure were predominantly cohesive in Biodentine, TheraCal and GIC groups while RMGIC showed majority of mixed and minority adhesive failures.

Conclusions: Biodentine demonstrated lower bond strength values when immediately bonded to bulk-fill resin composite compared with RMGIC, TheraCal and GIC groups

INTRODUCTION

For decades, the restorative management of dental caries involved the placement of a lining on the floor and, when present, axial walls of the

cavity.⁽¹⁾ The placement of a lining was proposed for several reasons: to reduce the number of viable bacteria remaining close to the pulp, to induce development of reactionary/reparative dentine, to possibly remineralize remaining demineralized

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hard tissues, to isolate the pulp against thermal and electric conduction, to protect pulpal cells against chemical irritants such as methacrylates from adhesives and to prevent the effects of restoration leakage on the pulp. ⁽²⁾

The traditional lining materials include calcium hydroxide, glass ionomer, resin modified glass ionomer, and pure resinous liners with particles releasing therapeutic agents. The success of liners underneath resin composite (RC) depends not just on the bond quality of the liner to the dentin, yet additionally on the nature of bond amongst liner and overlying RC. Various researches suggested the use of resin-modified glass ionomer cement (RMGIC) instead of GI in the sandwich technique because of improved bond strength to RC due to its chemical bonding. ^{(3),(4)} The bond quality of RMGIC to RC varies relying upon the kind of adhesive utilized and it has been demonstrated that self-etch is superior to total etch. ⁽⁵⁾

Extensive researches have been taking place in creating bioactive materials with a potential for remineralization. ⁽⁶⁾ Bioactivity refers to apatite-forming capability while biomineralization is the ability to get tied down to the dentin through formation of a mineral-rich interfacial layer and tag-like structure outspreading from the interfacial layer to the dentinal tubules. ⁽⁷⁾

The evolution of bioactive calcium silicate cement (Biodentine™ (BD) and TheraCal, etc.) set a landmark in the development of a unique category of materials combining bioactivity, biocompatibility, and strength. ⁽⁸⁾

Biodentine™ is recently being used as a dentine replacement material under composite restorations. A study by Hashem et al. showed no significant difference in bond strength between BD and RC in either self-etch or total etch mode. ⁽⁹⁾

Theracal is a new light-cured resin-modified calcium silicate-filled base/liner material designed for direct and indirect pulp capping. Theracal exhibited

physiochemical bonding to the dentin and is well-tolerated by immortalized odontoblast cells. ⁽¹⁰⁾

The utilization of bioactive liners underneath resin composite (RC) would clinically be more valuable than utilizing GI liners as they are biologically well-tolerated by the pulp tissue ⁽¹¹⁾ and have relatively higher re-mineralizing capability. ⁽¹²⁾

Conventional resin based composite (RBCs) would be light cured in 2mm thick increments of material. But, there is a demand to bulk cure RBCs in 4 to 6mm increments to reduce clinical procedure times. The increasing popularity of restorative materials – so-called “bulk-fill” materials – are claimed to enable restoration build-up in layers up to 6 mm thick. This new material class includes flowable and packable types. ⁽¹³⁾

Adhesive dentistry is a rapidly changing and evolving field. The basic principle of adhesion of composite resins to dental substrate is based on exchange processes in which inorganic dental material is replaced by synthetic resin. ⁽¹⁴⁾ Currently there is a tendency to simplify bonding procedures which introduced the self-etching adhesive concept. Recently, a new single bottle universal or multimode adhesive with silanes was introduced that simplifies the bonding procedure as single adhesive and can be used in self-etch or total etch or selective etch mode and on any surfaces (enamel, dentin, any direct, or indirect restorative materials) without additional primer. ⁽⁴⁾

The bond strength of liner to restorative materials has been an issue of concern. To our knowledge little/no study till now has compared the bonding ability between TLC, BD, and RMGIC to bulk-fill RC using universal adhesive. Hence, in the present study the shear bond strength (SBS) of BD/TLC/RMGI/ GIC to bulk-fill composite using universal adhesive was evaluated and compared and the null hypothesis was that there is no difference in the SBS within each substrate (TLC/BD/RMGI/GIC). The study also intended to categorize the specific modes of failure.

MATERIALS AND METHODS

The materials used, composition, manufacturer and mode of application are shown in (Table 1).

TABLE (1) Materials composition, manufacturer and mode of application

<i>Material</i>	<i>Material composition</i>	<i>Manufacturer-lot #</i>	<i>Mode of application</i>
<i>Tricalcium silicate cement - Biodentine</i>	Powder: di-, tri-Ca silicate, CaCO ₃ , Fe, and Zr oxides Liquid: H ₂ O, CaCl ₂ , and modified polycarboxylate	Septodont, St Maur-des-Fosses, France -B06211	Five drops of liquid added to the capsule. Capsule triturated at 4,000 rpm for 30s
<i>light-cured resin-modified calcium silicate cement - TheraCal</i>	45% wt type III Portland cement, 10% wt radiopaque component, 5% wt hydrophilic thickening agent (fumed silica) and approximately 45% resin	Bisco Inc, Schamburg, IL, USA 1700000367	Inject the material into cavity in 1mm layer – light cure 20 s
<i>Resin-modified glass ionomer cement (RMGIC)</i>	Powder:Fluoroaluminosilicate glass Liquid: H ₂ O, polyacrylic acid, HEMA,	Harvard Dental International GmbH Hoppegarten, Germany 7510294	Mixing premeasured capsule in amalgamator for 10 s. light cure 20 s
<i>Glass ionomer cement - GIC</i>	Powder: aluminosilicate glass Liquid: H ₂ O, polyacrylic acid, and tartaric acid	Medi-CEM; PROMEDICA Dental GmbH Hoppegarten, Germany - 1722569	Mixing powder/liquid ratio 1:1 Fill the cavity
<i>Bulk-fill resin composite</i>	Matrix; urethane dimethacrylate (UDMA) Triethyleneglycol Dimethacrylate (TEGDMA) Di- and trimethacrylate resins Carboxylate Filler; 85.5% w and %66.4V - silanated strontium aluminum sodium fluoride phosphate silicate glass	Quixfill Dentsply DETREY GmbH. Konstantz. Germany - 1509000951	Insert in single increment – light cure for 40 s
<i>Universal dental adhesive</i>	MDP phosphate monomer, dimethacrylate resin, polyalkenoic acid copolymer, filler , ethanol, water, initiator - Silane	Single Bond Universal™, 3M ESPE, St. Paul, MN, USA 502225	applied on liner surface with a bristle brush, rubbed for 20 s followed by gentle air drying with oil-free compressed air for approximately 5 s to evaporate the solvent and was light cured for 10 s

Tooth Preparation; Forty human intact premolars extracted for orthodontic reasons were collected for the study and the teeth were cleaned with ultrasonic scalers and stored in saline. These teeth roots were invested in acrylic resin blocks using a cylindrical mould that was 15 mm/25 mm in dimension. The occlusal surfaces were grinded

perpendicular to the long axis of the tooth with a high-speed diamond disc (KG Sorensen, São Paulo, SP, Brazil) to obtain a flat surface such that the occlusal surfaces were flush with the acrylic surface. Then a cavity of 4 mm diameter and 2 mm depth was prepared (Figure 1; a and b) to retain the liner using a highspeed handpiece with a cylindrical

carbide bur (56;KG Sorensen, São Paulo, SP, Brazil). The cavity dimensions were verified by a digital caliper (accuracy ± 0.01 mm). Carbide bur was changed every 3 preparations. These 40 samples were randomly divided into four groups (n=10/group): Group I - BD; (Biodentine™, Septodont, Saint-Maur-des-Fossés, Creteil, France), Group II - TLC; (Theracal LC™, Bisco Inc, Schamburg, IL, USA), Group III - RMGIC; (Harvard Dental International GmbH Hoppegarten, Germany) and Group IV- GIC; (Medi-Cem, PROMEDICA Dental, Hoppegarten, Germany) and the cavities were filled as per manufacturer's instructions [Table 1] and their surfaces were not finished to mimic the clinical scenario. Universal adhesive, (Single Bond Universal™, 3M ESPE, St. Paul, MN, USA) was applied on TLC/BD/RM GIC -GIC surface with a bristle brush, rubbed for 20 s followed by gentle air drying with oil-free compressed air for approximately 5 s to evaporate the solvent and then light cured for 10 s after placing the polyethylene tube (4-mm diameter, 5-mm height) as per the manufacturer's instructions. Bulk-fill RC (Quixfill, Dentsply DETREY GmbH, Konstanz, Germany) was placed in the tube (Figure 1; c) and light-cured with a light-emitting diode light-curing unit (LED 105 Monitex Industrial Co.,Ltd,China) with an intensity of 1,200 mV/cm² for 20 s. After the

completion of RC curing, the polyethylene tubes were removed with a sharp knife. All specimens were incubated at 37°C in water for 24 h.

Measurement of shear bond strength

A circular interface modified lap shear test was designed to evaluate the bond strength. All samples were individually and horizontally mounted on a computer controlled material testing machine (Model 3345; Instron Industrial Products, Norwood, USA) with a loadcell of 5 kN and data were recorded using computer software (Bluehill Lite; Instron Instruments). Samples were secured to the lower fixed compartment of testing machine by tightening screws through metallic custom-made housing device with central cavity into which the acrylic block fit (dimensions;25x25 mm). Shearing test was done by compressive mode of load applied at resin-liner interface using a metallic rod with half-circle shaped end attached to the upper movable compartment of testing machine traveling at cross-head speed of 0.5 mm/min (Figure d). The load required to de-bonding was recorded in Newtons.

Shear bond strength calculation; The load at failure was divided by bonding area to express the bond strength in MPa; $\tau = P / \pi r^2$ where; τ =shear bond strength (MPa, P =load at failure(N), π =3.14 and r =radius of composite disc(mm)

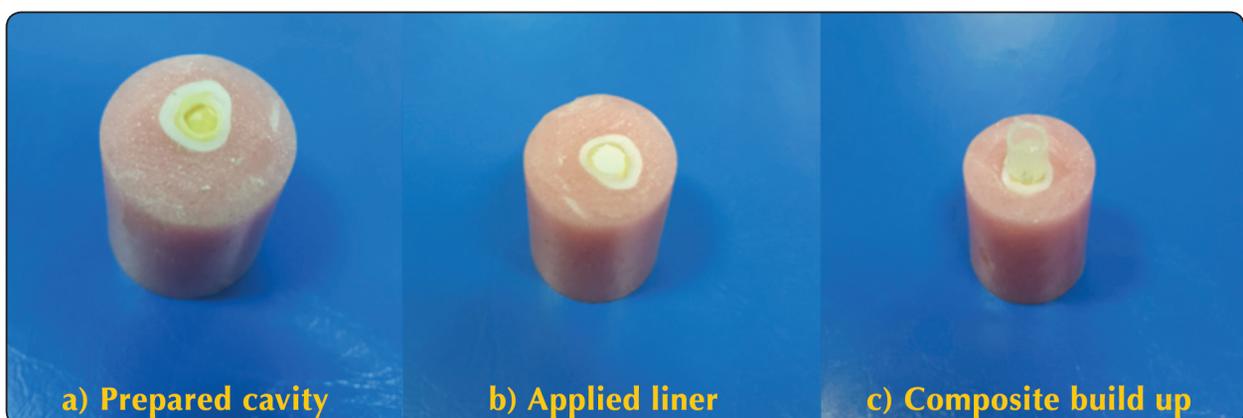


Fig. (1) Steps of shear bond strength sample preparation

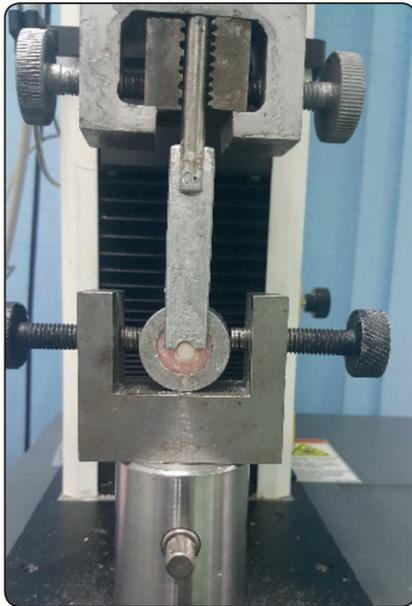


Fig. (2) Shear bond strength sample mounted onto testing machine

Failure analysis; The fractured test specimens were examined under a stereomicroscope (Leica MZ6, Mannheim, Germany) at a magnification of $\times 25$ and fractures were classified as follows: Cohesive failure - Failure within TLC/BD/RMGIC/GIC or RC, adhesive failure - Failure at RC- TLC/BD/RMGIC-GIC interface, and mixed failure - When two modes of failure occur simultaneously. Fracture analysis was performed by a single observer who was completely uninformed about the experimental groups.

Statistical analysis; statistical analysis was performed by using MS Excel 2010 and Asistat 7.6 statistics software for Windows (Campina Grande, Paraiba state, Brazil). Descriptive statistical data was presented in the form of mean and standard

deviation. Since a normal distribution was observed for all the bond strength values of all groups, one-way analysis of variance (ANOVA) was performed to assess the significance between the different groups followed by Tukey's *post hoc* tests were used for multiple group comparisons. *P* values ≤ 0.05 considered to be statistically significant in all tests.

RESULTS

The mean shear bond strength and standard deviation are shown in Table (2) and graphically drawn in figure (3:a). The highest shear bond strength mean \pm SD values were recorded with RMGIC group (6.962 ± 0.33 MPa) followed by TheraCal group (3.722 ± 0.74 MPa) then GIC group (3.491 ± 0.96 MPa) while the lowest shear bond strength mean \pm SD values were recorded for biodentine group (2.257 ± 0.68 MPa). One-way analysis with ANOVA revealed significant difference in bond strength values between the different groups ($p < 0.001$). Tukey's post-hoc test showed non-significant difference between (TheraCal and GIC) groups.

The observed modes of failure were predominantly cohesive in Biodentine group. In TheraCal and GIC the modes of failures were somehow similar with predominant cohesive failure and little adhesive or mixed failure while RMGIC showed majority of mixed and minority adhesive failures with no record for cohesive failure. Chi square test showed significant difference in failure mode distribution between the different groups ($p < 0.5$). table (2) and figures (3:b and 4)

TABLE (2) Comparison of shear bond strength results (Mean ± SD) and failure analysis (%) between all liner groups

Variable		Shear bond strength	Failure mode		
		Mean ± SD	Cohesive	Adhesive	Mixed
Liner group	Biodentine	2.257 ^C ± 0.68	8 (80%)	1(10%)	1(10%)
	TheraCal	3.722 ^B ± 0.74	5(50%)	2(20%)	3(30%)
	RMGIC	6.962 ^A ± 0.33	0(0%)	3(30%)	7(70%)
	GIC	3.491 ^B ± 0.96	5(50%)	3(30%)	2(20%)
Statistics	P value	<0.0001*	0.0207*		

Different letter indicating significant (p<0.05)

*; significant (p<0.05)

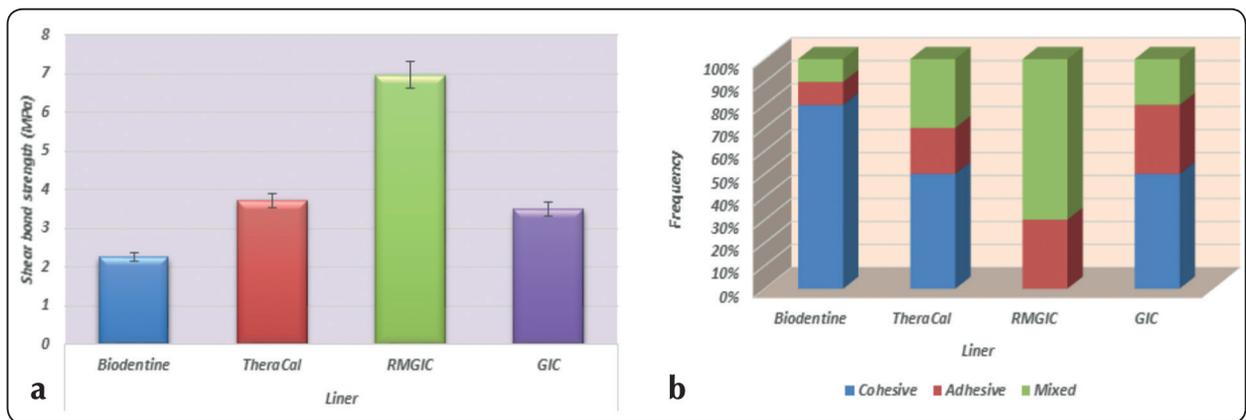


Fig. (3) Shear bond strength means values (3:a) and failure mode distribution (3:b) for liner groups

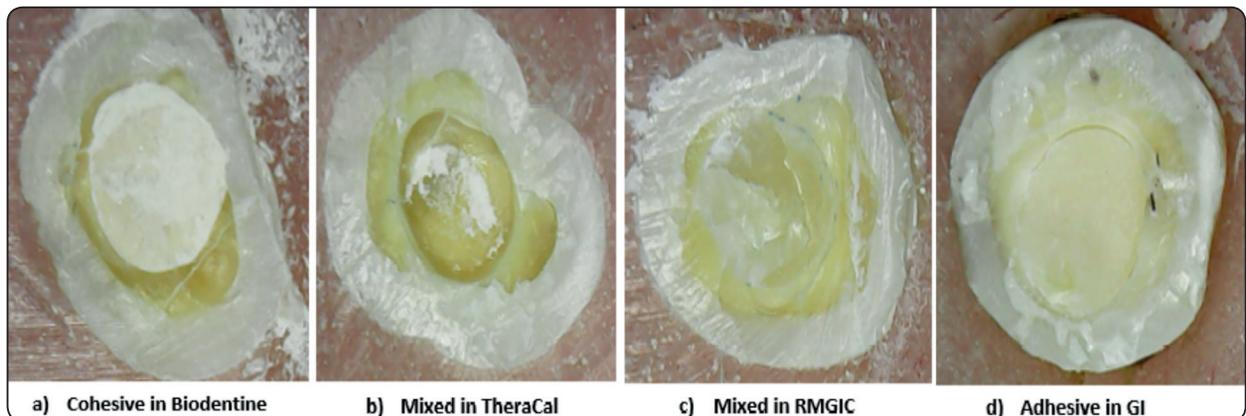


Fig. (4) Representative stereomicroscopic images for different failure modes within all groups (× 25)

DISCUSSION

Most of the bond strength studies are achieved using dentin pulp floor or axial walls as substrate. Studies on flat dentin surfaces are far from reality, since they do not take into account the clinical situation including presence of liner, resin insertion technique or stress induced in cavity geometry. In the present study, shear bond strength of Biodentine, TheraCal, RMGIC and Glass ionomer cement liners to bulk-fill composite was evaluated and compared.

Biodentine™ and TheraCal LC™ are calcium silicate-based bioactive liners that are proposed as alternatives to glass ionomers (GIs). Both materials release calcium and silicon ions into the underlying dentin.⁽¹⁵⁾ According to Saito et al. silica is a stronger inducer for dentin matrix remineralization than fluoride ions of RMGIC. Cytotoxicity studies showed that Biodentine/TheraCal is well-tolerated by immortalized odontoblast cells. These are the cells that retained their ability to divide with stable phenotypic protein expression profiles and ability to produce mineralized dentin extracellular matrix under *in vitro* conditions.⁽¹²⁾

Biodentine is a biocompatible bioactive material which may simulate dentine regeneration by inducing odontoblast differentiation from pulp progenitor cells and has been suggested to be used as a liner under resin composite restorations.⁽¹⁶⁾ It has higher compressive strength values than reinforced zinc oxide-eugenol cement, comparative performance to a resin modified GIC regarding microleakage when used as a dentine substitute⁽¹⁷⁾ and better marginal adaptation to dentine compared to MTA cement and GIC.⁽¹⁸⁾

In the present study, methacryloyloxydecyl dihydrogen phosphate (MDP)-based, universal adhesive with silanes was selected. This self-etch 10-MDP-based adhesive shows chemical bonding to Ca ions, and Al and zirconium oxides.^(19,20) The bifunctional silane molecule bonds chemically to silica-containing materials and has methacrylate

functionality that allows chemical union with resinous substrate. Silanes also act as adhesion promoters by enhancing the wetting ability of the adhesive system. This adhesive was selected in this study, aiming for additional chemical bonding with Ca releasing bio active liners.⁽²¹⁾

There are numerous methods for assessing the adhesion of a dental material to dentin, including tensile, shear, and push-out bond strength tests. In this study modified shear-lap was done based on its relative easiness, simplicity, inexpensive and reproducibility compared to tensile bond testing and to avoid friction effect that occurs in push out bond testing.⁽²²⁾

Materials intended for posterior bulk-filling placement can be applied in one increment up to 4 mm thickness, thus skipping the time-consuming layering process. Improved self-leveling ability, decreased polymerization shrinkage stress, reduced cuspal deflection in standardized class II cavities and good bond strengths regardless of the filling technique and the cavity configuration are reported.⁽¹³⁾

In this study, RMGIC group and TheraCal group showed significantly higher bond strengths than Biodentine group. This may be related to their resin contents that attain early cohesive strength on photo activation. Also, this might be due to similar resin chemistry promoting chemical adhesion with RC as proposed for RMGIC. Hydroxyethyl methacrylate (HEMA) incorporated into the TheraCal and RMGIC forms a chemical bond with the resin of the composite. Additional chemical union is due to copolymerization of unreacted methacrylate groups present in the oxygen-inhibited layer of TheraCal / RMGIC with those of composite resin.^{(23), (24)} The resin bonding agent intermixes with both composite and TheraCal /RMGIC by true chemical bonding to create a strong interface.

Biodentine group showed the least SBS means, which may have been due to low early strength of

the material per se and this was in agreement with previous studies.^(4,9) Biodentine is a porous material that needs at least 2 weeks' time for crystallization of hydrated calcium silicate gel to attain bulk strength adequate to withstand the polymerization stresses.⁽²⁵⁾ In the present study, bonding was performed to Biodentine immediately after 12 min to describe a single appointment clinical procedure. This might be the reason for low bond strength and cohesive failures in Biodentine.

Failure analysis (Figures 3:b and 4) revealed that higher bond strengths were often associated with 'mixed' or 'cohesive' failures in liner. Though the SBS of TheraCal and RMGIC groups were comparable, the failure modes were predominantly cohesive in TheraCal group while RMGIC group showed 70% mixed failures and 30% adhesive failures. Cohesive failure in TheraCal could have been due to its low bulk strength. TheraCal, a resin-modified (RM) calcium silicate cement is a combination of a HEMA/TEGDMA-based resin and calcium-silicate powder. On light activation, HEMA and TEGDMA monomers create a polymeric network able to stabilize the outer surface of the cement. Thus, formed poly-HEMA is hydrophilic and favors the absorption of moisture and triggers a second setting reaction that is hydration of calcium silicate particles with liberation of calcium ions.⁽⁷⁾ TheraCal releases more Ca ions than RMGIC. Hence, a chemical bonding among adhesive and Ca, Al, Zr, and silicon ions of TheraCal may have resulted in closer bond strength values as RMGIC group despite its low bulk strength.

CONCLUSION

Within the limitations of this *in vitro* study, the following conclusions might be drawn:

1. Biodentine demonstrated significantly lower bond strength values when immediately bonded to bulk-fill resin composite. The mode of failure was cohesive within Biodentine, indicating its weakness in its initial setting phase.

2. This necessitates paying attention towards the importance of leaving Biodentine for longer time to reach its final strength before the application of composite restoration.
3. RMGIC and TheraCal accomplished satisfactory bond strength to withstand condensation forces and stress from overlying composite resin due to the presence of a resin matrix.
4. Bulk-fill composite restoration can be placed immediately over TheraCal and RMGIC as alternatives to glass ionomers, completing the procedure in single visit
5. The interface integrity can be preserved by the composite insertion technique and by the type of liner used

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