

Comparative Evaluation of Effect of Polymerizable and non-Polymerizable Desensitizing Agents on Crown-Retentive-Strength of Zinc-Phosphate, Glass-Ionomer and Compomer Cements

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Abstract - The Purpose of this study was to evaluate the effect of polymerizable and non-polymerizable dentine desensitizers on retention of complete cast crowns cemented with three different types of cements. Freshly extracted human molars (n=90) were prepared for standardized crown preparation (6-degree taper, 4-mm height). The axial surface area of each preparation was determined and specimens were distributed equally among groups (n=10). Dentine desensitizers, cementing agents, glass ionomer cement and compomer cement. Teeth were prepared and individual castings were made using high noble porcelain-metal alloy. Castings were cemented, thermo-cycled and removed along the path of insertion using a universal testing machine. Tooth surface as well as inner surface of the casting was examined and nature of cement failure was determined. Compomer cement exhibited the highest retentive strength and all dentine treatments resulted in significantly different retentive values. Zinc phosphate was the least retentive. Crown retentive values of Compomer cement were improved with Prime & Bond NT and Gluma Desensitizer. Retentive values of zinc phosphate cement with Prime & Bond NT were decreased and not affected with Gluma Desensitizer. Retentive values of Glass ionomer cement were not affected by any of the desensitizers used in the study.

KEY WORDS: desensitizer agents, crown retention

INTRODUCTION

Dentine hypersensitivity is a common cause of pain that affects one in every seven dental patients¹. Frequently patients exposed to fixed restorative procedures experience discomfort in the prepared teeth either during the treatment or sometimes after placement of restoration, which they perceive in the form of pain or sensitivity^{2,3}. After tooth preparation, dentine can become sensitive as a result of microleakage and the resultant formation of bacterial byproducts². Hydrodynamic theory speculates that whenever a pain-producing stimulus is applied to the external tooth surface, the fluid flow within the dentinal tubule in either direction stimulates the sensory nerves in dentine or pulp and the open tubules allow fluid flow leading to sensitive dentine³. The treatment of hypersensitive teeth should therefore be directed towards occluding the tubules so as to limit fluid movement. The restorations luted with cements like zinc phosphate and glass ionomer result in partial removal of the smear layer owing to their acidic nature. The cement can be forced into the patent tubules before it sets⁴. Cement can displace an equal amount of dentinal fluid, which may cause excessive hydrostatic pressure leading to post-cementation sensitivity. Before cementation the orifices of the exposed tubules can be sealed off with polymerizable products like dentine

bonding agents or with non-polymerizable formulations, which do not require light activation. The geometric form of the prepared tooth & the casting mainly determines the retention of the prosthesis, but the clinical success of the restoration also depends on the integrity and the mode of attachment of the intervening cement layer⁵. Application of dentine desensitizers alters the physical and chemical characteristics of prepared tooth surface and thus can increase or decrease the retentive ability of the cements. The purpose of this study was to evaluate and compare the influence of two different types (polymerizable and non-polymerizable) of dentine desensitizers on the retentive ability of two routinely available [zinc phosphate (ZP), glass ionomer (GI)] and one recently developed [compomer (CM)] luting agents. The purpose of selecting the ZP and GI cement is that they have a broad spectrum of uses in prosthodontic practice and their retentive ability following application of desensitizers is still confusing. The newly developed CM cement was evaluated first time for its retentive ability after application of the desensitizers.

MATERIALS AND METHODS

Standardized tooth preparation

Ninety freshly extracted molars were selected by measuring the width in the range between 11-13mm (at the level of cement-enamel junction) with the help of a digital Vernier-Caliper. Teeth were cleaned of surface debris, sterilized for a short term in 0.5% sodium hypochlorite, and then stored in water. Non-carious unrestored molars were selected, and

* MDS

roots were notched and embedded in a U shaped cross sectioned Aluminium block (25mm X 10mm X 10mm) using clear autopolymerizing resin (Orthodontic Resin; Dentsply Caulk, Milford, DE), with the cemento-enamel junction positioned 1 mm above the top of the ring. At all times after mounting, the teeth were stored in sterile water, which was changed frequently. A new device was designed for standardized tooth preparation which clamps the high-speed air-rotor hand piece in such a position that the long axis of the bur remains parallel to the analyzing rod of the surveyor (Fig. 1A). The clamped hand piece maintains the long axis of the diamond point absolutely vertical all the time during preparation. Two custom made jigs (one for occlusal reduction and another for axial reduction) were designed to hold the mounted tooth specimen vertically and to adjust the level separately for occlusal and axial preparation. The occlusal surface was sectioned flat 5 mm above the top of the Aluminum block. Constant taper of 6 degrees for the preparations was obtained as a negative image of a round end tapered diamond point (Fig. 1B)⁶. The axial surface was reduced to a depth of 1 mm and axial length of 4 mm, using a new diamond point for each tooth⁷. A wheel shaped diamond point (WR- 13, Dia-Burs, Mani Inc, Japan) was used to prepare the occlusal surface. Axial reduction was achieved by using the long round ended tapered diamond point (TR-26, Dia-Burs, Mani Inc, Japan). The stable clamped handpiece and convergence of the diamond instrument automatically ensured a constant taper of 6 degrees no matter in which direction the holding jig was moved on the surveyor platform.

Die, wax pattern, casting fabrication:

Impressions of the prepared teeth were made using a simultaneous dual-mix technique with a polyvinyl-siloxane impression material (Exaflex, GC Corp, Tokyo, Japan) poured with Type IV gypsum (Ultrarock; Kalabhai Karson, Mumbai, India) to fabricate the dies. Two coats of die spacer (Heartman dental lab, Germany) and then the die

lubricant (Heartman dental lab, Germany) was applied on the master die. The die was subsequently dipped twice in molten wax (Dura Dip Wax; Belle de St. Claire, Chatsworth, CA) to achieve a consistent thickness of 1 mm. A wax-ring (3-mm diameter) was fabricated and attached to the centre of the wax pattern. The wax-patterns with rings were sprued and invested with a phosphate-bonded investment (Deguvest CF; Degussa AG, Hanau, Germany) according to manufacturer's instructions. The test crowns were cast using a high noble metal content porcelain-bonding alloy (Olympia; Jelenko Dental Products, Armonk, NY). The internal surface of each crown was airborne-particle abraded with 50-mm aluminum oxide particles at 60 psi (4.22 kg/cm²) (Omega Micro Pencil Air Abrasive Unit; APM Sterngold, Attleboro, Mass). The internal surface of all castings was cleansed using a steam cleaner at 70 psi (Williams Model, VMG-1.5-W; Williams Dental Co Inc, Amherst, NY) to remove remaining debris.

Desensitizer application

The perimeter of the occlusal surface of the preparation was measured by adapting 1.2-mm diameter lead foil closely along the midlevel of the axial surface of the prepared tooth as shown in Figure 2. The foil was cut where the end overlapped and the length of the foil was measured. This dimension for each tooth was multiplied by the axial length (4 mm) to arrive at the axial surface area of each preparation. The contribution of occlusal region would be minor since they were not thought to develop an adhesive bond to dentine and casting. Thus the values of stress could be exaggerated by incorporating the occlusal surface area into the calculation⁸. Preparations were ranked by decreasing surface area and distributed into nine subgroups so that each cementation group contained similar mean surface areas. Thirty samples were treated for each group prior to cementation: 1) No application (Control group) 2) Prime & Bond NT application 3) Gluma desensitizer application. The control treatment consisted of crowns cemented on



Figure 1A. Standardized preparation unit on the surveyor platform.

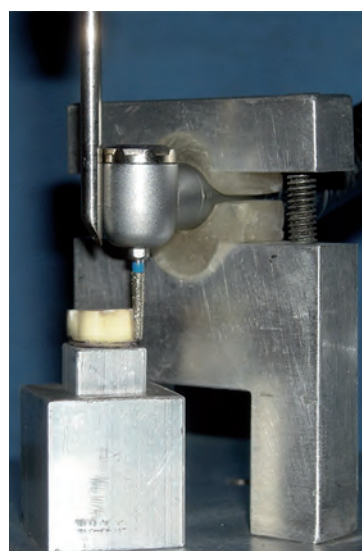


Figure 1B. Standardized preparation of mounted extracted teeth.

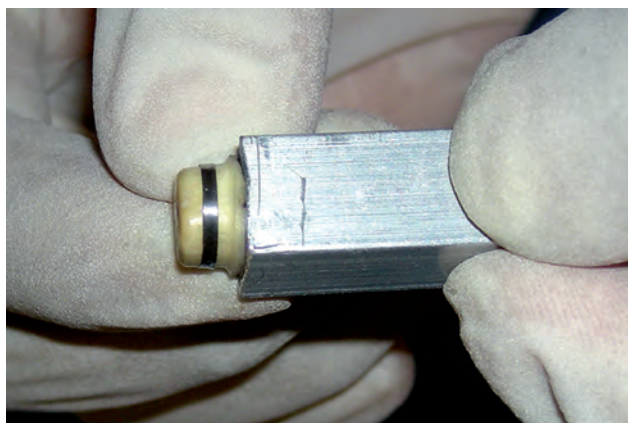


Figure 2. Perimeter of the occlusal surface of the preparation was measured by adapting 1.2-mm diameter lead foil closely along the midlevel of the axial surface of the prepared tooth.

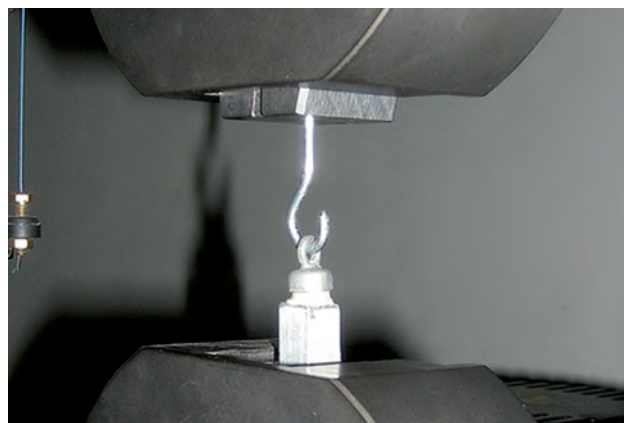


Figure 3. Restorations removed along the path of insertion using an universal testing machine at the crosshead speed of 0.5 mm/min.

prepared teeth without use of a desensitizer. Thirty samples were treated with the application of the Prime & Bond NT which is fifth-generation (one-component) dentine bonding agent marketed by the Dentsply/Caulk Company. According to the manufacturer, it is distinguished from other “one-component” bonding agents in that it contains extremely small (7-nanometer) filler particles. The dentine was etched with 34% phosphoric acid gel for 15 seconds. Thereafter the preparations were rinsed for 10 seconds and moisture was removed so that the dentine remained visibly moist. The Prime & Bond NT Adhesive was applied for 20 seconds, air dried for 5 seconds and light activated for 10 seconds (LED Light curing machine; AT&M Biomaterials, Beijing, China). Thirty samples were treated with the application of the Gluma desensitizer. It is non-polymerizable glutaraldehyde based dentine sealer. It is composed of an aqueous solution containing 5% glutaraldehyde, 35% 2-hydroxyethyl methacrylate (HEMA) and purified water. Glutaraldehyde is a biologic tissue fixative that coagulates and denatures the serum proteins in the dentinal tubule fluid. Hydroxyethyl methacrylate is water soluble that facilitates the deep penetration of glutaraldehyde into the dentinal tubule. HEMA molecule is an analog to methyl methacrylate, except that the pendent methyl ester is replaced by an ethoxy ester group to make it hydrophilic. A smallest possible amount of Gluma Desensitizer was applied on the prepared tooth surfaces using applicator tip for 30-60 seconds. Then a stream of compressed air is applied until the fluid film has disappeared and the surface is no longer shiny and rinse thoroughly with water.

Cementation

ZP was mixed on the glass slab in a powder to liquid ratio of 1 gm: 0.5 ml as recommended by the manufacturer. The first increment was incorporated into the liquid in 20 seconds. The other increments of powder were mixed, each for 15 seconds using a circular motion and covering a wide area of the slab to allow dissipation of exothermic heat. The powder to liquid ratio for GI (Fuji-I) as recommended by the manufacturer for luting procedures is 1.8 g: 1.0 g. One level of scoop of powder and two drops of liquid were taken every time as recommended. All the

powder was rapidly incorporated into liquid and mixed for 20 seconds. Two scoops of CM powder (approx. 0.25 gm) were dispensed onto the mixing pad. Two droops of CM liquid (approx. 0.12 gm) were dispensed onto the mixing pad. The cement was mixed for 30 seconds to reach the ideal luting consistency which flows 3/4”-1” from the spatula. Uniform layer of mixed cement was applied on the internal surface of the coping and seat on to the prepared tooth. Due to the dual-cure property of CM, the operator has the option of utilizing a curing light to facilitate clean up. Immediately after seating is verified, excess cement can be briefly light-cured at the margins by constantly moving the curing light tip around the margins for no more than 10 sec. Excess cement will reach the “gelled” stage after this 10 seconds cure. All excess cement will remain in the gelled state for approximately 45 seconds. All excess cement was removed before final self-cure set was achieved. Following all excess removal, exposed margins were be light cured 20-40 seconds to assist restoration stabilization.

Manufacturers’ directions were followed in mixing all 3 cements. Prepared teeth and castings were paired for cementation. In order to achieve a constant film thickness of the cements, it was imperative to mix the cements according to manufactures instructions and also to maintain a constant seating force during cementation. The crowns that were lined with cement were initially seated with strong thumb pressure. Thereafter, the assembled teeth and crown were placed on the customized spring loaded device (at a load of 5 kg) for 5 minutes. The excess cement was removed after set using an explorer. Twenty-four hours after cementation, the crowns were cycled between water reservoirs at 5°C and 55°C for 2500 cycles, using a 30-second dwell time at each temperature. Crowns were subjected to an axial dislodgment force until failure on a universal testing machine (Model TTMBL; Instron Corp, Canton, OH) at a crosshead speed of 0.5 mm/min (Fig. 3).

The force at dislodgment and nature of debonding were recorded. Immediately following casting dislodgement, 2 observers independently examined the casting and tooth without magnification to arrive at the category of failure based on the criteria given in Table I. When a difference existed between examiners, a consensus was achieved

Table 1. Categories for characterization of type of failure after crown removal

Category	Description
1	Cement principally on tooth (>3/4 axial surface)
2	Cement on both casting and tooth
3	Cement principally on casting (>3/4 axial surface)
4	Fracture of tooth root without casting separation

Table 2. Comparison of three groups of combination of cementing agents in different desensitizers by 2-way ANOVA followed by Newman Keuls post hoc procedure

Cementing agents	Group- C		Group-P		Group-G	
	Mean	Std.Dev.	Mean	Std.Dev.	Mean	Std.Dev.
ZP	4.51	0.27	2.88	0.44	4.48	0.34
GI	5.60	0.28	5.47	0.38	5.35	0.35
CM	6.00	0.15	7.06	0.18	7.65	0.31
F-value	111.9655		399.2972		262.4839	
p-value	0.0000*		0.0000*		0.0000*	
Pair wise comparison by Newman Keuls post hoc procedure						
ZP-GI	0.0001*		0.0001*		0.0001*	
ZP-CM	0.0001*		0.0001*		0.0001	
GI-CM	0.0017*		0.0001*		0.0001*	

*p<0.01

Table 3. Comparison of three desensitizers in different groups of combination of cementing agents by 2-way ANOVA followed by Newman Keuls post hoc procedure

Desensitizers	ZP		GI		CM	
	Mean	Std.Dev.	Mean	Std.Dev.	Mean	Std.Dev.
Group-C	4.51	0.27	5.60	0.28	6.00	0.15
Group-P	2.88	0.44	5.47	0.38	7.06	0.18
Group-G	4.48	0.34	5.35	0.35	7.65	0.31
F-value	74.4924		1.5726		151.8328	
p-value	0.0000*		0.2241		0.0000*	
Pair wise comparison by Newman Keuls post hoc procedure						
C-P	0.0001*		0.6199		0.0001*	
C-G	0.9778		0.1961		0.0001*	
P-G	0.0001*		0.6861		0.0001*	

*p<0.01

Table 4. Results showing the percentage of castings by failure type. The bolded values indicates the predominate failure modes

Technique	Category (As per the Table 1)			
	1	2	3	4
ZP cement	10	50*	40	00
ZP cement + Prime & Bond NT	00	00	100*	00
ZP + Gluma Desensitizer	00	10	90*	00
GI cement	20	60*	10	10
GI cement + Prime & Bond NT	50*	40	10	00
GI cement+ Gluma Desensitizer	60*	30	10	00
Compomer cement	40	10	30	10
Compomer cement + Prime & Bond NT	30	10	20	40
Compomer cement + Gluma Desensitizer	30	00	10	60*

Pearson's chi-square=66.1224, df=24, p=0.0000

*Predominant failure

by re-examination. Dislodgement force was converted to stress using the axial surface area calculated for each preparation. The data for dislodgment stress were analyzed using a 2-factor ANOVA. Given significant main effects, the Newman Keuls post hoc procedure was used for comparison of multiple pair-wise mean values. The data for characterization of the type of failure was analyzed using the nonparametric chi-square test of association for independent specimens. All statistical testing was conducted at the 95% level of confidence.

RESULTS

CM cement exhibited the highest retentive strength and all dentine treatments resulted in significantly different retentive values [Gluma (7.60 ± 0.58 MPa) > Prime & Bond NT (6.97 ± 0.35 MPa) > control (6.00 ± 0.32 MPa)] (Table II and III). Retention of GI was between the CM and ZP group: control (5.60 ± 0.49 MPa) = Prime & Bond NT (5.47 ± 0.70 MPa) = Gluma (5.35 ± 0.82 MPa). ZP had the lowest retention values: control (4.51 ± 0.42 MPa) = Gluma (4.48 ± 0.58 MPa) > Prime & Bond NT (2.88 ± 0.76 MPa). Graphs (Fig. 4, 5) depict the above mentioned data.

The results for characterization of failure mode are presented in Table IV. For castings cemented with ZP-control group, cement was found both on casting and tooth surface (Fig. 6A), but the mode of failure of ZP (with both desensitizers) was found to be predominantly at cement-tooth interface as the cement was only found on the casting (Fig. 6B,C). The mode of failure of GI-control group, cement was found both on casting and tooth surface (Fig. 6D). The cement was predominantly retained on the tooth surface with the castings cemented with GI after application of both types desensitizers (Fig. 6E,F). The CM cement without application of desensitizer predominantly remained on tooth surface (Fig. 6G), however with application of both the desensitizers, cohesive tooth fracture was observed (Fig. 6H,I).

Polymerizable desensitizer (Prime & Bond NT) used in this study is a fifth generation bonding agent which shows resin tags penetrated inside the dentinal tubules, adhesive layer present over the surface and Hybrid layer (interdiffusion zone) in between them as shown in Scanning Electron Micrograph (Fig. 7A). Scanning Electron Microscopic analysis of the Gluma desensitizer treated dentine revealed that the majority of tubules were obliterated with a thin layer that covered the surface and infiltrated into tubules as plugs (Fig. 7B).

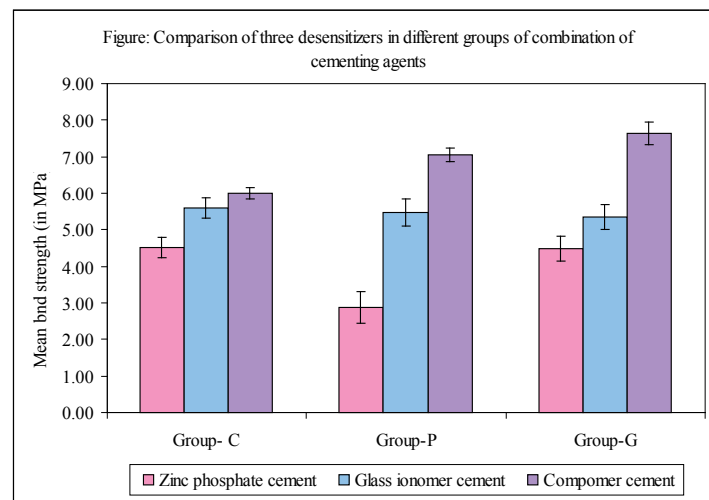


Figure 4. Comparison of desensitizers with different cementing agents.

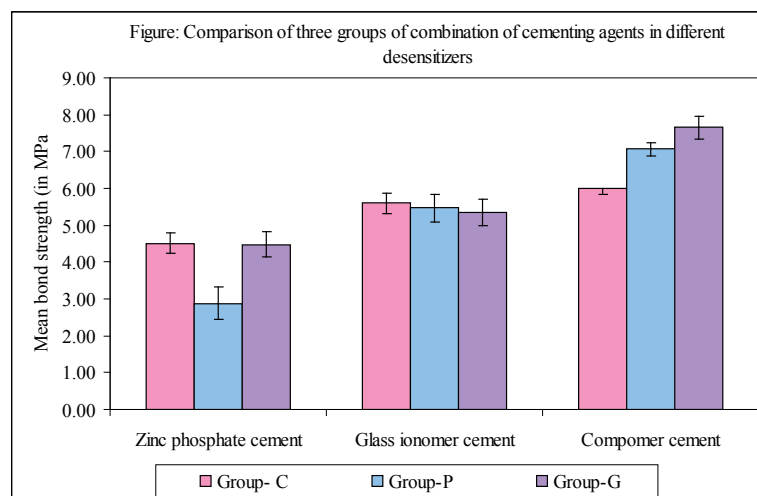


Figure 5. Comparison of cementing agents with different desensitizers.

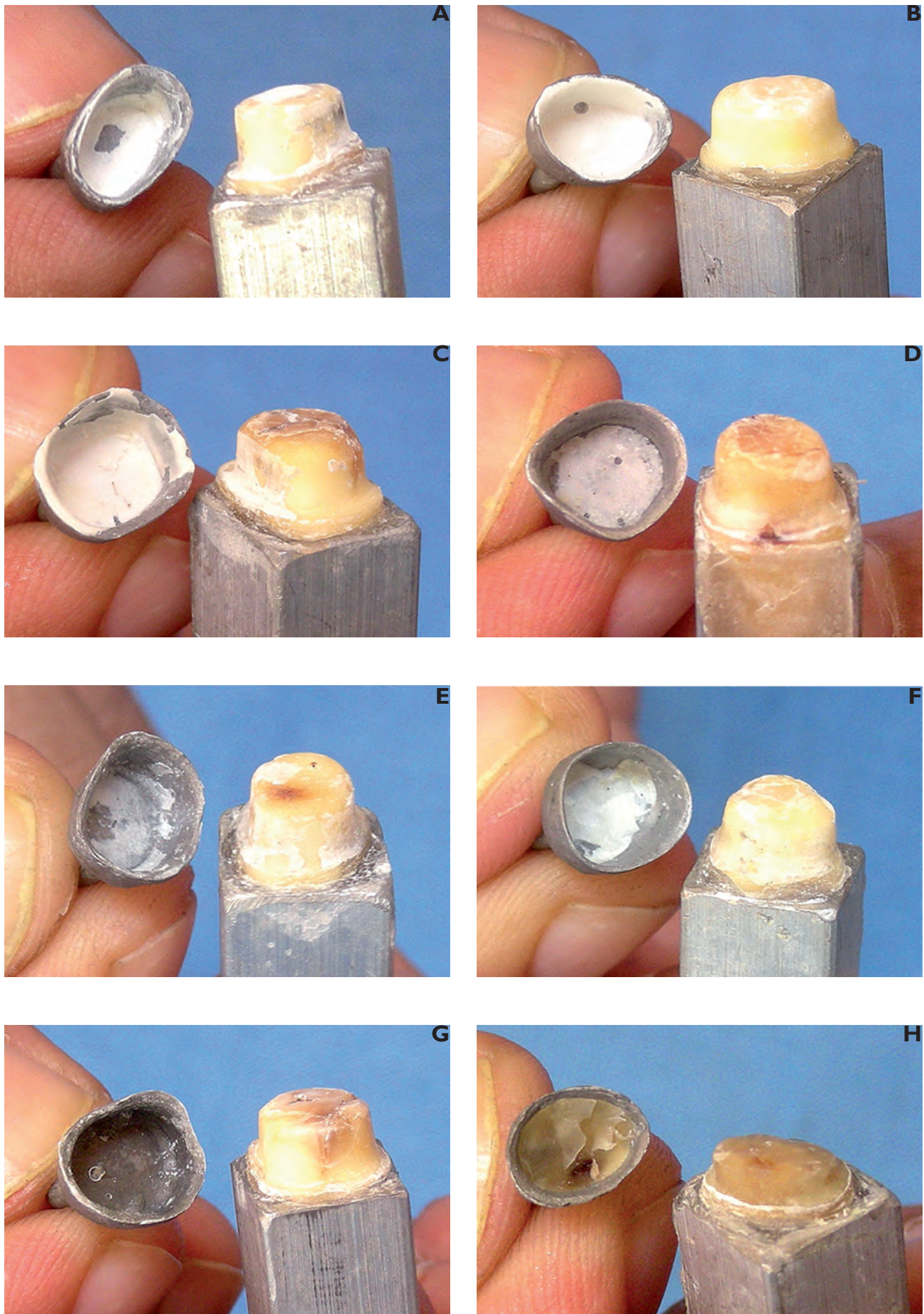


Figure 6. Examples of predominant types of failures for each cement-desensitizer group: ZP cement without desensitizer (A), with Prime and Bond NT (B) and with Gluma Desensitizer (C); GI cement without desensitizer (D), with Prime and Bond NT (E) and with Gluma Desensitizer (F); and CM cement without desensitizer (G), with Prime and Bond NT (H).

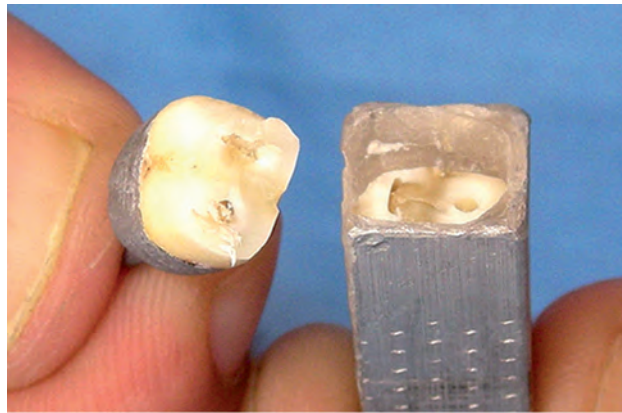


Figure 6 I. Examples of predominant types of failures for each cement desensitizer group: CM cement with Gluma Desensitizer.

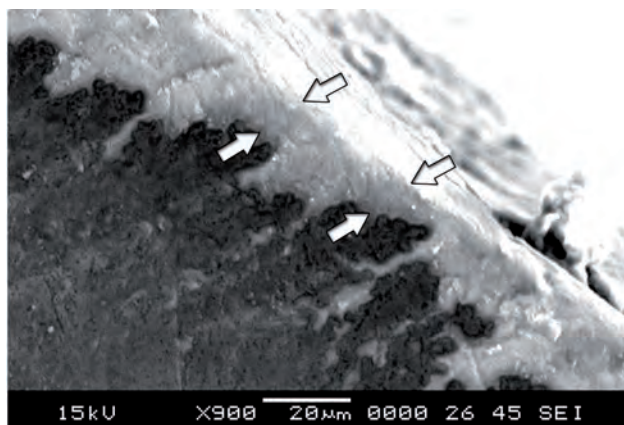


Figure 7A. SEM Micrograph of Gluma Desensitizer treated dentine. A thin layer seen over the dentine (between arrows), occluding the entrance of the tubules.

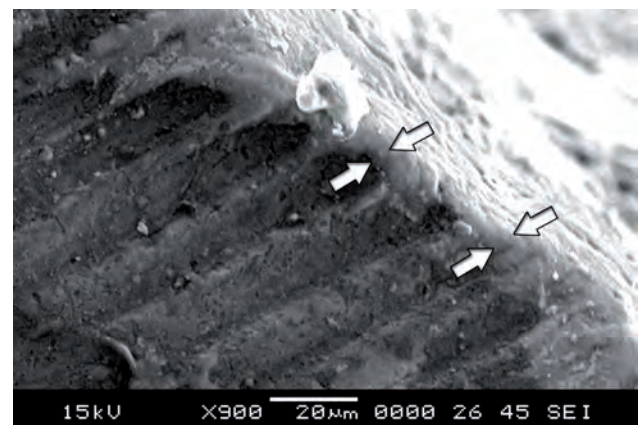


Figure 7B. SEM Micrograph of Prime & Bond NT treated dentine, comparatively thicker layer occluding the tubules (between arrows).

DISCUSSION

Previous studies show confusing results of effect of polymerizable dentine desensitizers as well as non-polymerizable desensitizers on crown retentive values when luted with ZP or GI cements^{7,8}. The retentive value of CM cement influenced by any type of desensitizer is yet to be studied. Hence this study was performed using “Prime & Bond NT” & “Gluma desensitizer” as “polymerizable” & “non-polymerizable dentine desensitizer” respectively. The cementing agents used were ZP, GI & CM. Use of various adhesive resin cements were extensively studied previously for their retention values with entire range of desensitizers hence not included in the study. The effect of the application of dentine desensitizers on crown retentive values of different cementing agents can be explained with two hypotheses. First hypothesis is “bond strength of the cement agents rely on the mechanical interlocking into surface irregularities”. Second hypothesis is “use of dentine desensitizers that have the ability to chemically react with the cement will provide enhanced crown retention”. When relying mainly on cementing agent for crown retention, the order of retentive strength of cements was CM cement > GI cement > ZP cement, despite of any desensitizing agent.

For ZP group the Prime & Bond NT significantly reduced the crown retention to low levels, compared with the

control, while retentive value did not affected significantly with the Gluma Desensitizer. ZP cement attains its retentive qualities by filling irregularities in the prepared dentine and the internal surface of casting⁹. The Gluma Desensitizer may reduce some of the dentine surface irregularities that aid in mechanical retention for ZP cement but casting retention nevertheless unaffected. Gluma Desensitizer infiltrates into the tubules as plugs and form thin layer over the dentine occluding the entrance of the tubules which may not affect surface irregularities much (Fig. 7A); hence the casting retention was found to be unaffected when compared with control group.

For the GI group the use of any type of dentine desensitizer did not affect the crown retention significantly compared with control. Recent developments in the areas of GI cements, namely their chemistry and in vitro bond strengths, have many characteristics in common with the development of the fifth generation adhesive systems¹⁰. This may be the reason that retentive value of GIC was not decreased significantly in spite of relatively smoother surface formed by application of Prime & Bond NT (fifth generation adhesive).

For the CM group both types of dentine desensitizers significantly increased crown retention. CM cement had significantly greater retentive value with application of Gluma

Desensitizer than Prime & Bond NT. Four samples in Prime & Bond NT and six samples in Gluma Desensitizer group with CM cement demonstrated overt root fracture without crown separation. Teeth demonstrating root fracture failed at higher values than those teeth demonstrating crown separation. The strength of root dentine is 50-90 MPa^{11,12}. Cohesive root fracture is a result of excessively high-stress concentration resulting from complex geometry in the embedded specimen. This geometry leads to simultaneous shear and tensile loading that opens a dentine crack in a small region at an average stress of 6.97 MPa and 7.60 MPa, but at micro-localized stress of >100 MPa.

Higher values of retentive strength were recorded for CM cement with and without dentine desensitizer compared to ZP or GI cement with or without desensitizers. Hamid A et al (1998)¹³ found the release of Hydroxyethyl methacrylate (HEMA) from light activated GI and CM cement. Relatively high and rapid diffusion of HEMA through dentine indicates that free unpolymerized HEMA was present at the restorative-dentine interface, even after polymerization of the cements. This released HEMA may have high chemical affinity towards the HEMA present in the Gluma Desensitizer indicating greater retentive strength values. With application of Prime & Bond NT, CM cement shows significantly increased retentive strength value, may be because of the chemical affinity of the polymerizable resin monomer towards the HEMA present in the CM cement. According to the manufacturer, Gluma desensitizer is composed of an aqueous solution containing 5% glutaraldehyde, 35% 2-hydroxyethyl methacrylate (HEMA) and purified water. Glutaraldehyde is a biologic tissue fixative that coagulates and denatures the serum proteins in the dentinal tubule fluid. Hydroxyethyl methacrylate is water soluble that facilitates the deep penetration of glutaraldehyde into the dentinal tubule. Arris CAG et al¹⁴ studied effect of desensitizing agents on dentinal tubules and showed that thin layer can be seen over the dentine, occluding the opening of dentinal tubules when treated with Gluma Desensitizer. In the clinical study the primer of the original Gluma Adhesive system (currently marketed as Gluma Desensitizer), the desensitizing solution was applied to crown preparations¹⁵. The authors concluded that Gluma primer reduce sensitivity through a protein denaturation process with concomitant changes in dentine permeability. This theory recently has been supported by studies using confocal microscopy¹⁶, which found formation of transversal septa occluding the dentinal tubules following application of Gluma desensitizer. SEM analysis in this study also found that Gluma Desensitizer infiltrates into the tubules as plugs and form thin layer over the dentine occluding the entrance of the tubules confirming the results of above mentioned studies.

Reductions in sensitivity may result from formation of resin tags and a hybrid layer when a dentine bonding agent was used as a polymerizable dentine desensitizer¹⁷. Mode of failure of cementing agents supports the results regarding the variations in crown retentive values. With application of desensitizers, ZP cement predominantly remained on the castings which indicates that the retentive ability of the cement with tooth surface decreased drastically especially with Prime & Bond NT. Because of pattern of deposition on the dentine as well as the manufacturers' instructions of application (Gluma desensitizer

needed to be applied in smallest possible amount) the thin layer of Gluma desensitizer may not affect the surface irregularities as much as Prime & Bond NT causing changes in retentive values with ZP cement. GI cement (with or without application of desensitizer) shows mixed type of failure with adhesive tendency more towards tooth. This supports the results with non-significant effect on crown retentive values. As far as CM cement is concerned most of the samples (60% with Gluma desensitizer, 40% with Prime & Bond NT) shows cohesive tooth failure without restoration separation. This proves the results with significantly increased retentive values of CM cement with the desensitizers.

The effect of desensitizers on fluoride releasing ability of the GI cement as well as CM cement needs to be studied as the dentine sealing ability of desensitizers may affect this important property. The remnants of temporary cement also can be one of the factors which can affect the retentive values of permanent cementing agents. Temporary cement can be one of the parameters to be concerned in such type of studies. The limitations of these study in spite of absolute internal control are: 1) The environment of oral cavity cannot be optimally duplicated in vitro. 2) Crown retentive values observed here may be influenced by laboratory conditions. 3) A clinical set up presents with number of factors that create a different environment. Factors like operator variability in tooth preparation, restoration fabrication, temporization, crown cementation should be considered.

CONCLUSION

1) Prime & Bond NT and Gluma Desensitizer were found to be effective in improving retention of cast crowns when used with CM cement. 2) Crowns retentive values of ZP cement with Prime & Bond NT were decreased and not affected with Gluma Desensitizer. 3) Retentive values with GI cement were not affected by any of the desensitizers used in the study.

ADDRESS FOR CORRESPONDENCE

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